

METALLURGIA

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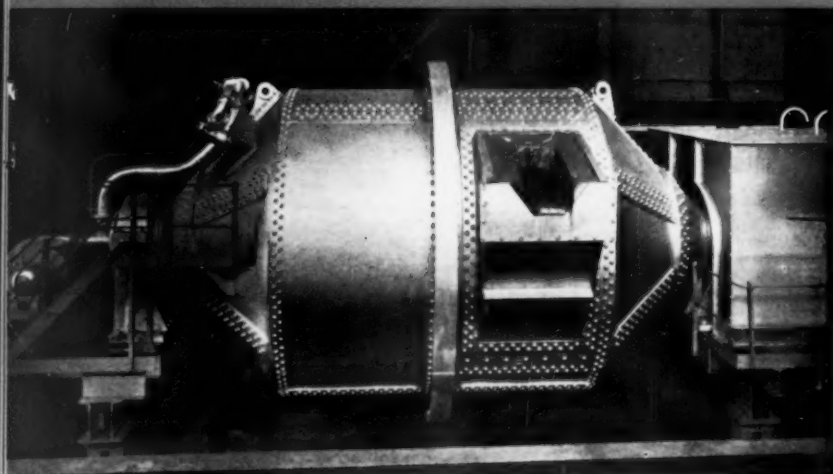
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Below A 150 ton Hot Metal Receiver supplied to Bombay Steel Works Limited.



Steel Works Plant

NEWTON CHAMBERS are now better equipped than ever before to offer a comprehensive service to the steel industry. Extensions to our constructional shops with the provision of new equipment enable us to make even larger and more intricate fabrications.

We bring to the service of the steel industry years of specialist experience in the design and manufacture of steelworks plant and equipment. In fact today we are one of the largest suppliers of steelworks ancillary plant, including ladles up to 300 tons capacity.

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THERMOCOUPLE ALLOYS

"Ni-Cr (plus) v/s Ni-Al (minus)"

Conforming to B.S. 1827:1952

The Thermo-electric properties of the Nickel-Chromium/ Nickel-Aluminium Alloys "Ni-Cr(Plus)" and "Ni-Al (Minus)", are in strict conformity with those stated in the American National Bureau of Standards Circular 561, and British Standard Institution Reference Tables for Thermocouples, B.S. 1827:1952.

For Compensating Cables and Extension Leads in Civil and Military Aircraft, "Ni-Cr(Plus)"/"Ni-Al(Minus)" Wires have Ministry of Supply (Air) type approval, for Cables manufactured to Specifications EL. 1705 and 1632.

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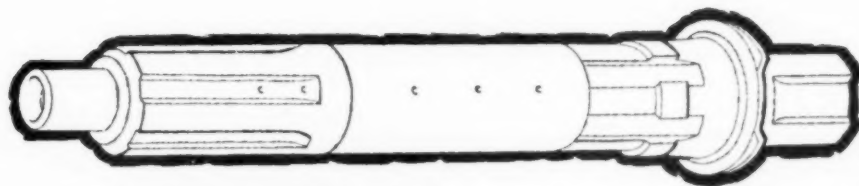
When ordering Compensating Leads from your usual supplier, for greater accuracy specify: "Thermocouple Wires made by A. C. Scott & Co. Ltd., preferred".

A. C. SCOTT & COMPANY LIMITED

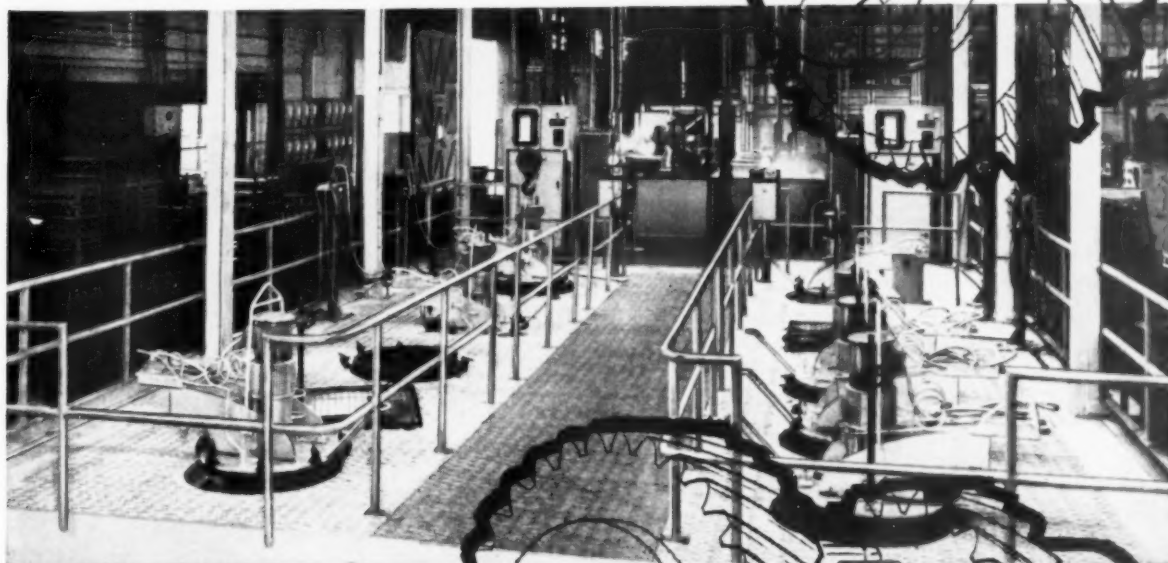
TECHNICAL ALLOY MANUFACTURERS FOR OVER THIRTY YEARS

Roundthorn, Wythenshawe, Manchester 23

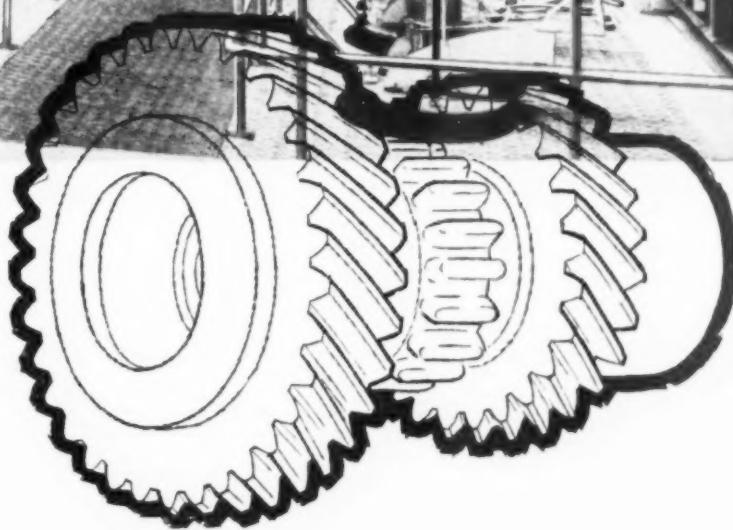
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Stainless Steel Strip Annealing Furnace

The illustration shows a Furnace for continuous treatment of ferritic or austenitic steel strip. Installed at the Stocksbridge Works of Samuel Fox and Co. Ltd., Sheffield.

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F144

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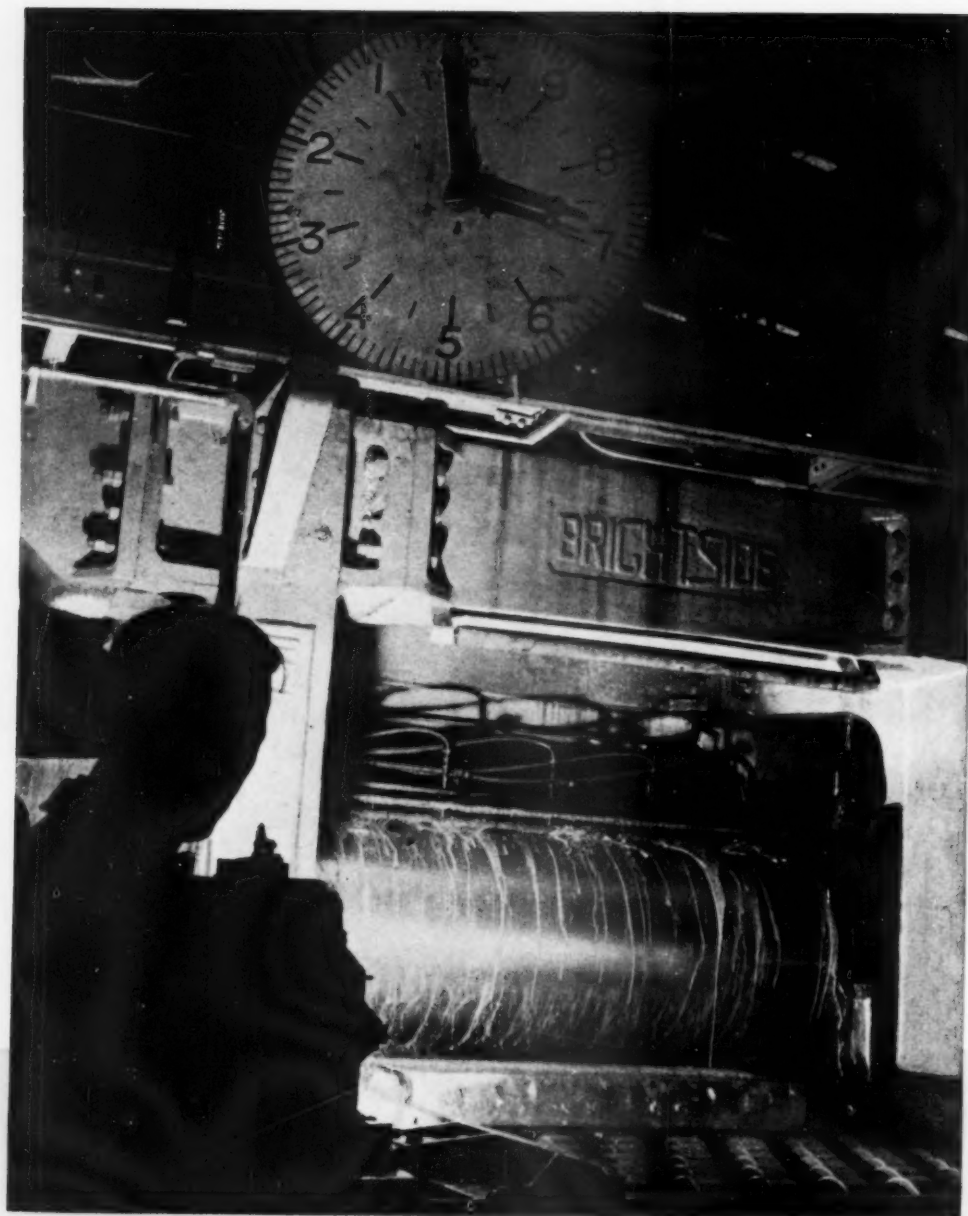
The Thompson L'Hospied service embodies much more than the production of accurate and consistently fine heat-resisting castings. Through their close association with the furnace industry they have first-hand knowledge of its requirements and unequalled facilities for testing under actual conditions.

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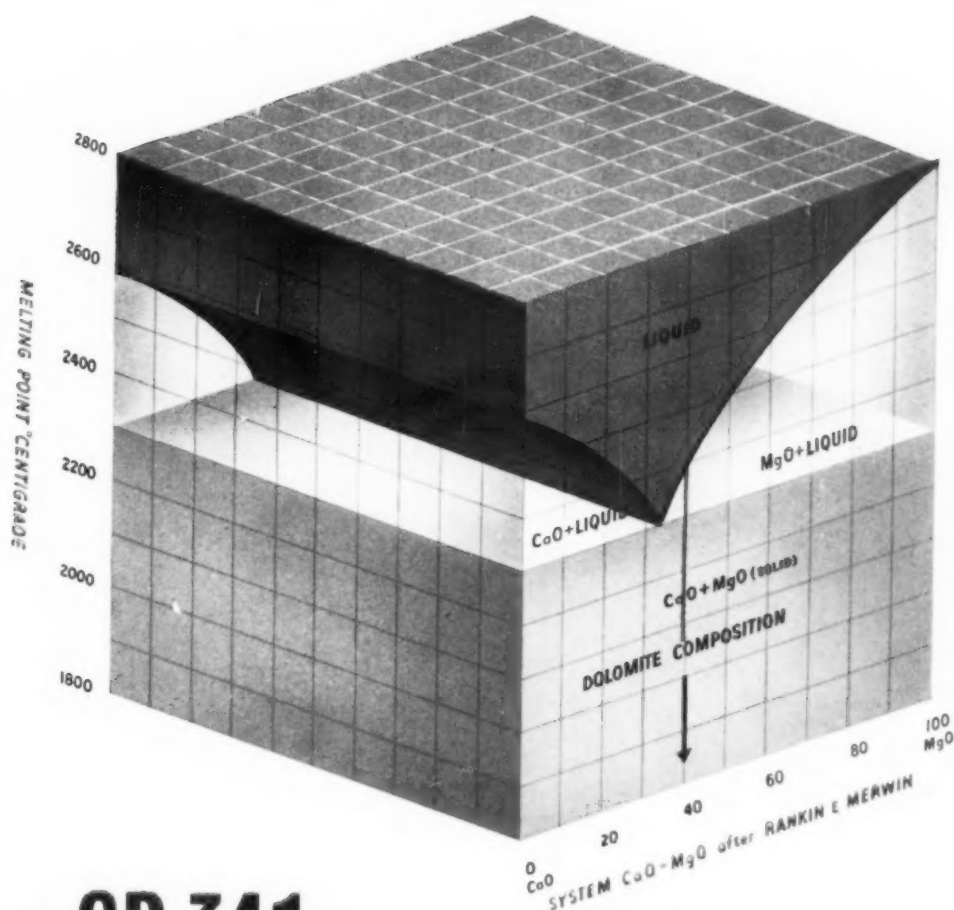
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Important News

for the Basic Steel Industry



New GR 341 Dolomite Brick Process

Dolomite an indigenous raw material relatively low in cost, is widely distributed in Great Britain. Its high melting point fits it for use in many metallurgical processes. Dolomite is the backbone of the basic steel industry. General Refractories have developed a new process of making bricks from this unique raw material, and they offer the "341" Dolomite brick to the steel industry for use in a wide range of applications.



GR Technical Service Department is available for consultation by users in the selection and application of these and other refractories.

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Basic O.H. Furnace sub-hearths, bottoms and checkers

Electric furnace side-walls and bottoms

KAL-DO
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ROTOR } Oxygen-Steel Converters

Active mixer sub-hearths and banks

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High performance

Low cost

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Philips EM 75B is a simply operated electron microscope with resolving power of 50Å specially designed to supplement optical microscopy in the range of 1200 to 3000 diameters. This instrument gives magnification up to 12,000 diameters on to photographic plates, making a further magnification to 120,000 diameters possible by enlargement.

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AUTOMATIC DEW POINT RECORDERS AND CONTROLLERS



Signal Controller

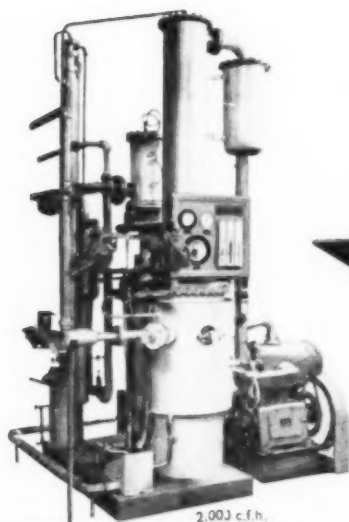


Recorder Controller

Autocarb systems automatically adjust the flow of air or enriching hydrocarbon to the furnace or generator in direct response to the dew point of the atmosphere. Specified conditions are automatically maintained. Autocarb systems are simple compact units which indicate, record and control the dew point of gas atmospheres in continuous or batch furnaces and atmosphere generators. They can control dew points to within plus or minus two degrees. On continuous furnaces separate zones of atmosphere control can be established and automatically maintained. If manual control is preferred, the Autocarb line includes simple recorders or indicators. On batch furnaces the Autocarb system will maintain the desired carbon potential for each temperature established in the heating chamber. On endothermic gas generators Autocarb systems automatically and continuously compensate for fluctuations in the composition of the reaction fuel gas supply and the humidity of the reaction air. This keeps constant the dew point of the product gas.

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Atmosphere generators with a wide range of applications in many industries. In the metal industry for bright annealing, brazing, sintering, gas carburising, bright hardening, etc.

GmvL Ammonia Cracker

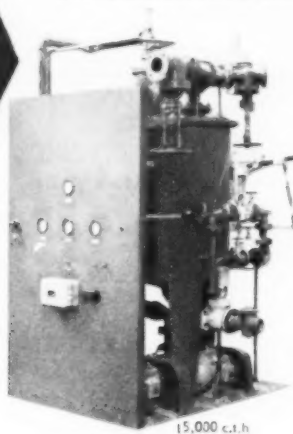
For bright treatment of stainless acid resisting and heat resisting steels, and for brazing and sintering generally.



150 c.f.h.

Gibbons Sub-X Inert Gas Generator

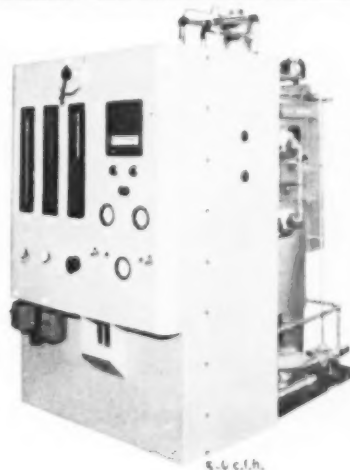
A highly efficient and compact producer of inert gas, operating on standard gaseous fuels and distillate grades of fuel oils.



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For gas carburising, dry cyanide treatment, etc. Automatic response to variations in demand while maintaining correct gas/air ratio.



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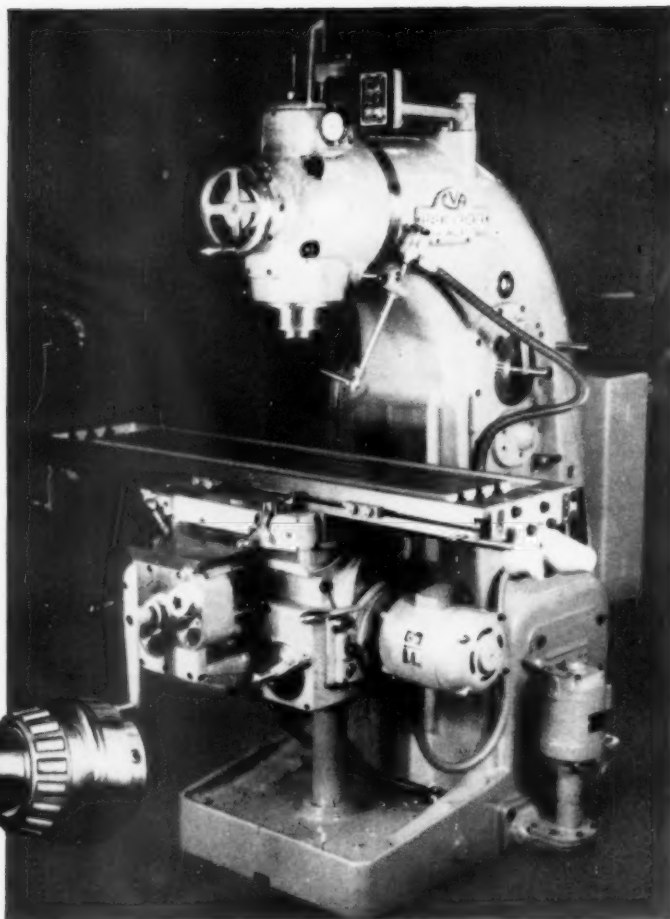
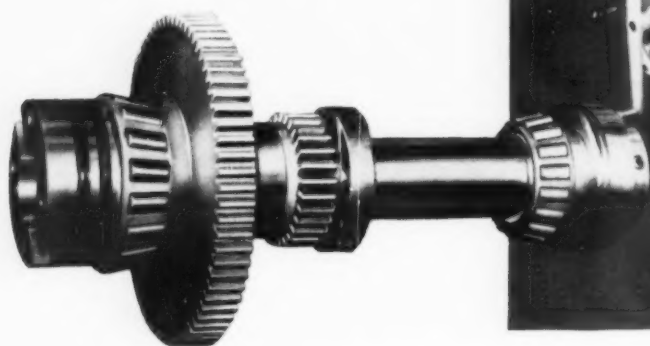
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NICKEL ALLOY STEELS

**ensure
reliability in
machining**



In the current designs of C.V.A. Jigs Moulds & Tools Ltd., Hove, who concentrate on high speed automatic and centre lathes, dicing presses and "Milwaukee" milling machines, the constructional materials include various nickel alloy steels for the highly stressed components. The 1½ per cent nickel-chromium-molybdenum steel, EN 24, one of the most useful compositions, is used for cams, shafts, spindles and other items which are either made from forged material or bar stock.

The reliability of C.V.A. automatics, now operating in thousands throughout the world, is a reflection of the reliability of nickel alloy steels under sustained stresses.

TYPICAL MECHANICAL PROPERTIES OF

EN 24

These steels exemplify its versatility both as regards strength and range of serviceable section sizes.

SIZE	HEAT TREATMENT	YIELD STRESS t.s.i.	MAXIMUM STRESS t.s.i.	ELONGATION per cent	IZOD ft. lb.
0.564" dia.	Oil quenched 830 C. tempered 200 C.	111.4	125.5	14.5	24
1½" dia.	Oil quenched 850 tempered 510 C.	79.5	83.6	15	28
1½" dia.	Oil quenched 850 C. tempered 560 C.	68.3	73.5	19	55
2½" dia.	Oil quenched 840 C. tempered 650 C.	57.3	64.1	20.5	65
5½" dia.	Oil quenched 830 C. tempered 650 C.	50.1	58.9	19.5	75

By utilising the better properties obtainable in more highly alloyed nickel steels, dimensions can be reduced, lighter constructions produced, distortion through heat treatment minimised and reliability and economy achieved.

Send for "The Mechanical Properties of Nickel Alloy Steels" ▶

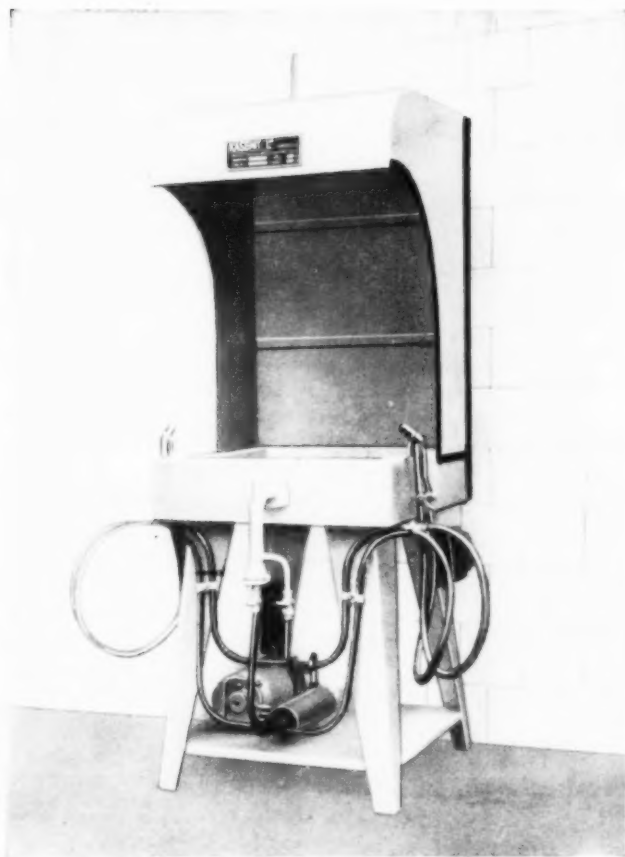
MOND NICKEL



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TGA 21/2/6



GAS BLAST COMBINED FORGE AND BRAZING HEARTH

Complete with Motor Driven Positive Air Blower, two Gas Blast Blow Pipes and one Gas Blast Burner. Suitable for Tube bending up to 1 in. diameter.

Size of Hearth, 20 in. × 20 in. × 4 in.

Gas Consumption, 200 cu. ft. per hour maximum.



NATURAL DRAUGHT GAS FIRED SALT BATH SUITABLE FOR CYANIDE OR NEUTRAL SALTS

Suitable for temperatures up to 900° C.

Obtainable in the following sizes :

8 in. dia. × 8 in. deep, gas consumption 300 cu. ft./hr.

8 in. dia. × 10 in. deep, gas consumption 350 cu. ft./hr.

10 in. dia. × 12 in. deep, gas consumption 400 cu. ft./hr.

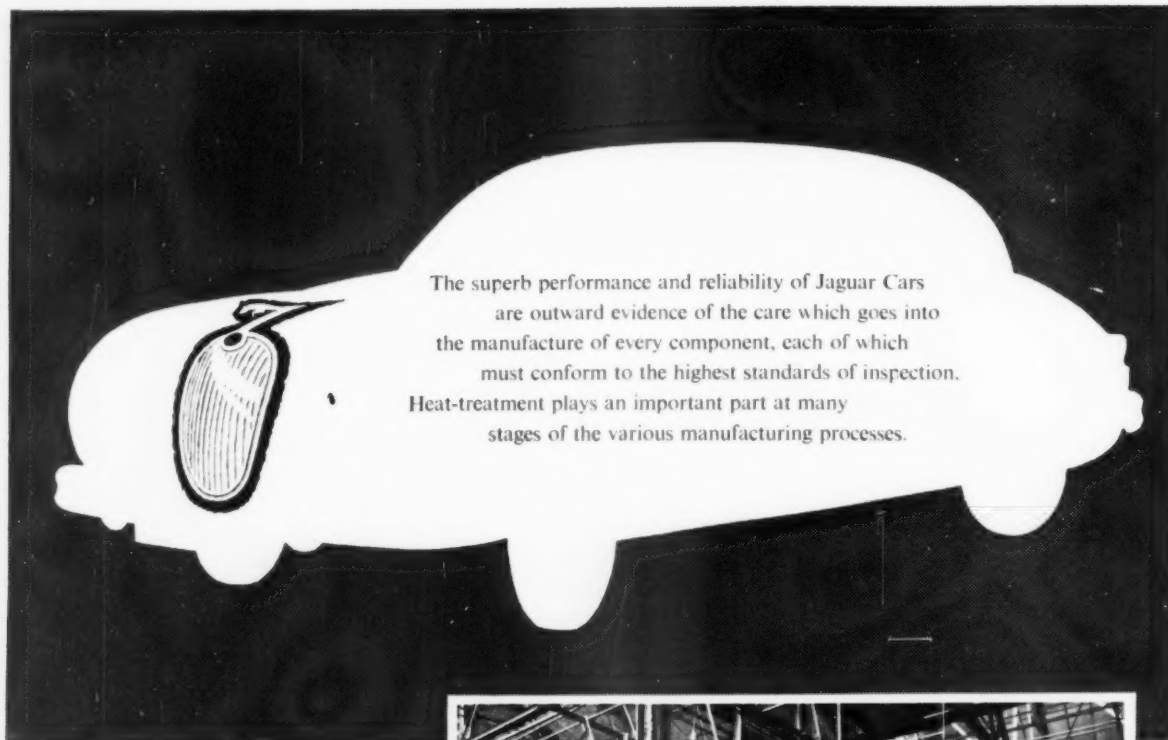
Time to heat up—1½ hours.

Manufactured by



7, HOLYROOD STREET · BERMONDSEY · LONDON, S.E.1

JAGUAR AND G.W.B.



The superb performance and reliability of Jaguar Cars are outward evidence of the care which goes into the manufacture of every component, each of which must conform to the highest standards of inspection. Heat-treatment plays an important part at many stages of the various manufacturing processes.

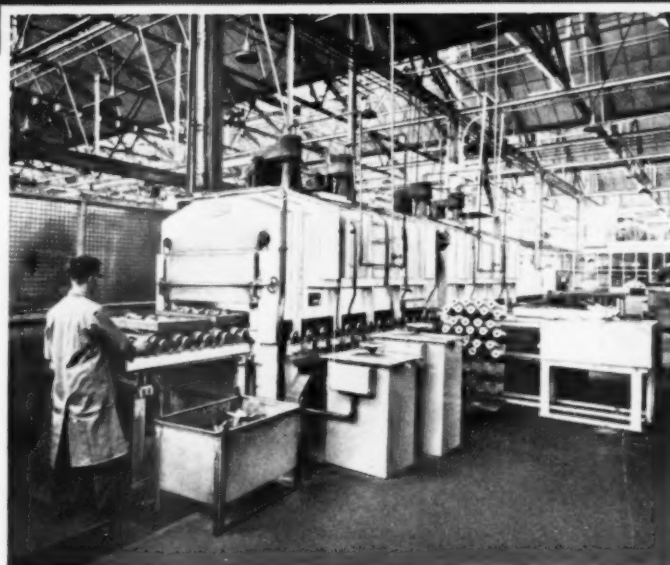
The latest addition to the heat-treatment shop at Jaguar Cars Limited is a G.W.B. electrically heated Roller Hearth Furnace specially designed to carry out normalising, cyclic annealing and stress relieving a variety of components.

SPECIAL FURNACE DESIGN

The unusual multi-purpose requirements have resulted in an installation in virtually two sections. (1) High temperature (950 C) furnace, followed by (2) forced draft cooling section, and finally, (3) a 650 C heating chamber.

APPLICATIONS

- Normalising: Welded components slow cooled from 900 C.
- Cyclic annealing: Gear forgings heated to 950 C, cooled to 550 C and re-heated to 650 C.
- Stress Relieving: Partly machined forgings such as crank shafts and gear blanks stress relieved between 550 and 650 C, and cooled to 200 C in the final 8' 6" long cooling chamber.



Over 25 years experience in electric furnace design at your disposal.



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Telephone: Dudley 55455 (9 lines)

Associated with: Gibbons Bros. Ltd., and Wild-Barfield Electric Furnaces Ltd.

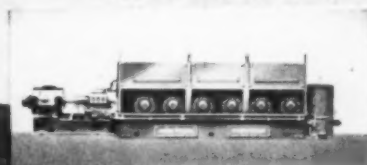
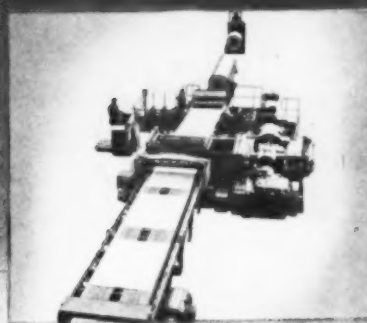
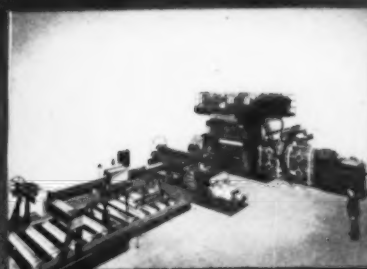
GWB 238

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Straightening Machines	— 390
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Wire Drawing Machines	— 215

Robertsons' products are continually improving in design and production are today well placed to the forefront in the new Atomic Age.



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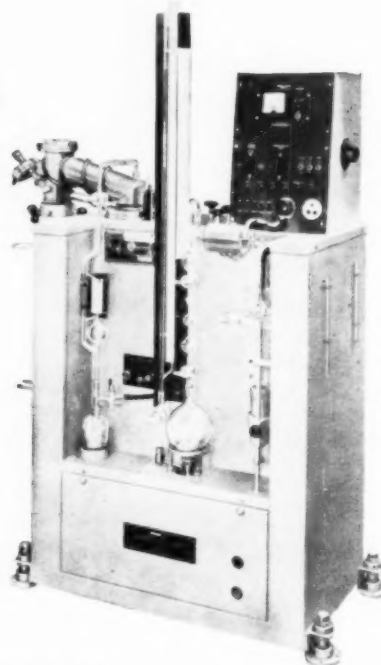
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**A Robust Unit for the Accurate
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- ★ Compact and robust cabinet with glassware protected by recessed front panel.
- ★ Samples up to 10-15 g. of steel can be accommodated.
- ★ Gas quantities of less than 10^{-5} g. can be detected.
- ★ 2M4 mercury diffusion pump with high compression ratio ensures large mass of gas can be extracted for sampling.
- ★ Small collecting space employed thus reducing a source of additional outgassing.
- ★ Overall size (without generator) 42 in. x 18 in. x 60 in. high.
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Picture of a man . . .



. . . with all the answers on insulating bricks

He is finding that the use of insulating bricks for more effective heat insulation has been a subject well worth looking into. Reading further he'll see that Newalls make a range of insulation bricks under the trade names "Nonpareil" and "Newparex" which are designed to cover heat insulation requirements up to 2012° F (1100°C). But why not read the catalogue yourself? Like the man in the advertisement, you'll find factual answers to all your queries.

*Write to us for a copy of our
INSULATING BRICK CATALOGUE
which will be supplied free on request.*

Newalls

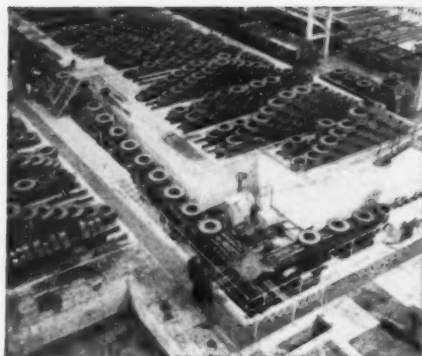
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P.O. BOX 50, THE ICKLES, ROTHERHAM, YORKSHIRE

HOT ROLLED STEEL STRIP

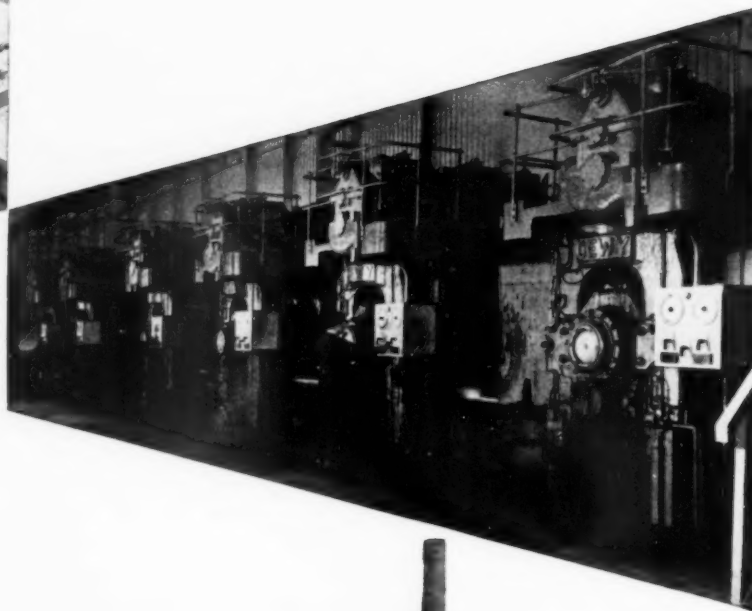


The new Brinsworth mill hot rolls steel strip

- in widths from 6 to 18 inches
- thicknesses from .042" to .250" varying according to width
- qualities which include mild steels, medium and high carbon steels, "Fortiweld," "Cor-Ten" and other special steels.

The Templeborough Strip Mill produces hot rolled strip

- in widths up to 6 inches
- thicknesses from .030" to .192" varying according to width.



SP 218



Vacuum melted steels are consistently free of inclusions and exceptionally clean. Steel for ball bearings treated in a vacuum furnace led to the following remarkable results:

1. Bearing life consistently 100%—200% better than usual standards for normal temperature operations.
2. Bearing life consistently up to 600% longer for high temperature, high speed applications.
3. Up to 90% reduction in bearing race rejects.

Would metals with such characteristics make your production better? Wild-Barfield—NRC vacuum melting furnaces enable you to develop materials specially suited to your needs. Backed by the experience of the National Research Corporation, who have built and operated more high vacuum furnaces than any other company in the world, Wild-Barfield—NRC vacuum equipment covers most needs. Write for details of the range.

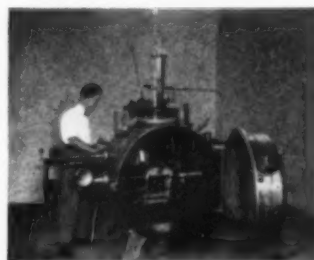
WILD-BARFIELD—NRC EQUIPMENT INCLUDES:

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VACUUM RESISTANCE FURNACES
VACUUM ANALYSING EQUIPMENT
HIGH VACUUM DIFFUSION PUMPS
LABORATORY VACUUM FURNACES

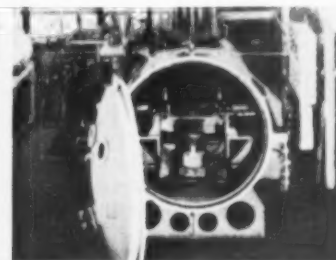


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**COULD A VACUUM FURNACE
DO THE SAME
FOR YOUR PRODUCTS?**



Model 2555 Vacuum Induction Furnace with melting capacity of 50 pounds of steel. Other standard furnaces have capacities of 12 to 3,000 pounds.



Model 2705 Non-Consumable Arc Skull Furnace with a capacity of 50 pounds of titanium. Other standard vacuum arc furnaces have capacities of 8 to 10,000 pounds of titanium.



for all heat-treatment purposes

WILD-BARFIELD ELECTRIC FURNACES LIMITED

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WB 96



METAL-CUTTING OILS

Oils, like metals, are difficult to illustrate . . . and to illustrate only a fraction of the immense variety we manufacture would require several pages in colour. Therefore, we will concentrate on telling you that we make a range of cutting oils blended to meet every requirement of the modern machine shop . . . for ferrous metals (including special hardened steels) and for non-ferrous metals.

Cutting oils formerly "good enough for the job" are just *not* so today. This age of specialisation demands that only the most suitable should be used. This is

sound common sense—and wise economy, too. So-called "cheap oils" are expensive at any price meaning more man-hours per job and higher scrap wastage.

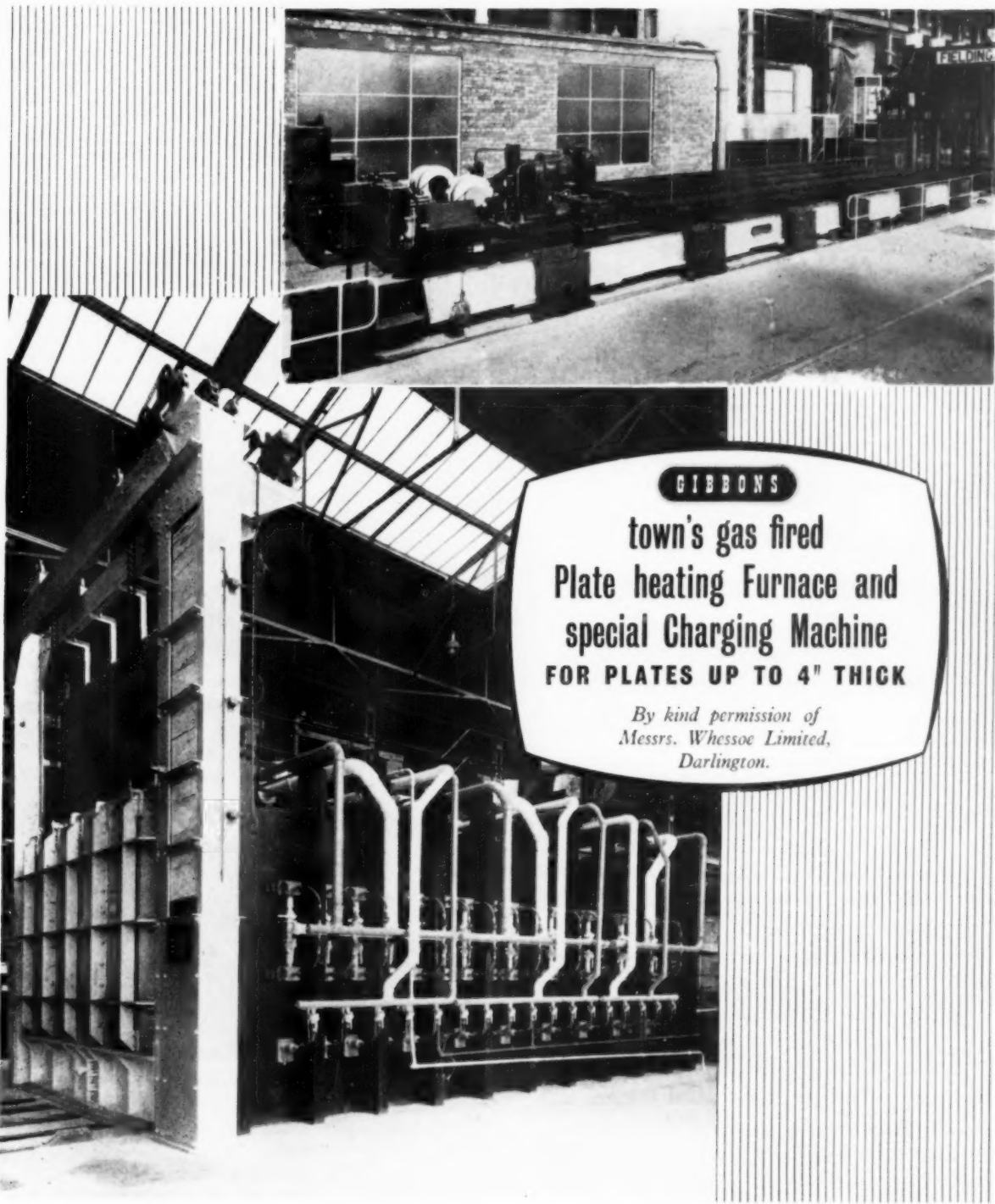
Keep your metal-cutting costs DOWN and productivity UP. Contact us before you start the job. Our organisation, backed by 60 years experience, is ready to assist you on all machining problems.

Send for descriptive brochure



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GIBBONS

town's gas fired
Plate heating Furnace and
special Charging Machine
FOR PLATES UP TO 4" THICK

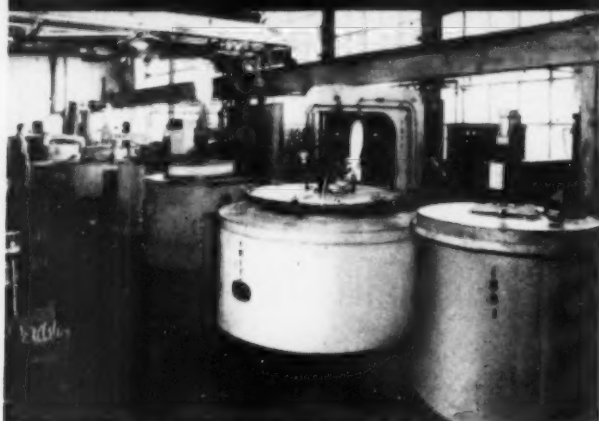
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*the choice
of the motor
manufacturer...*

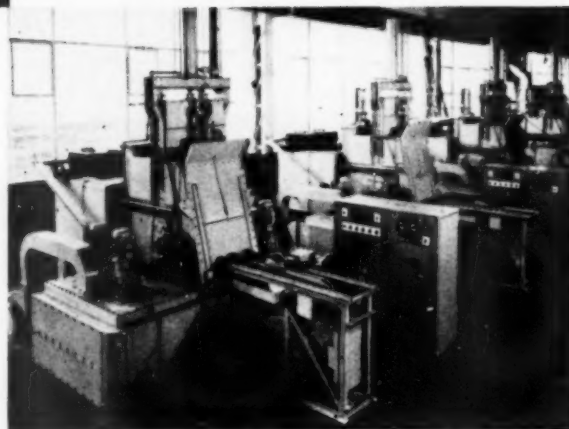


An installation of pit type furnaces with separate cooling chambers and oil and water quench tanks for carburising and hardening motor gear box parts.

Austin, Ford, Humber, and other leading motor manufacturers chose Efco Furnaces for hardening, gas carburising or carbonitriding gear components.

When you decide on Efco furnaces you may pick from the best of the World's current designs. For carburising and carbonitriding there are both vertical and horizontal closed quench furnaces, and vertical furnaces with separate cooling chambers. For hardening there are many continuous designs including shaker hearth, roller hearth and pusher furnaces.

EFCO furnaces



Humber Ltd chose Efco-Lindberg horizontal closed quench furnaces for carburising and carbonitriding gear components. The furnaces are electrically heated with Cor-therm elements.



BEST OF THE WORLDS
CURRENT FURNACE DESIGNS

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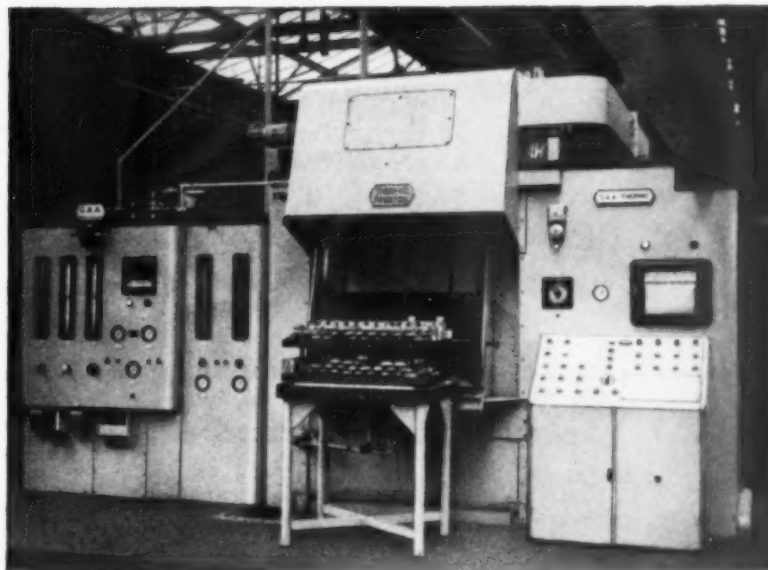
Heat Treatment

—Gas Board Demonstrates latest developments.

IN this modern general metals age of ours it seems a far cry to the days when the heat treatment of metals was considered to be a 'black art' and when the closely guarded secrets of the swordmakers and workers in precious metals were handed down from father to son. Today we recognise the value of the comparatively new technology of metallurgy and realise the benefits our civilization receives from the new metallic alloys and from the new plants and methods of processing these alloys which have been developed during the last few decades.

The type of plant we have in mind includes such items as the new heat treatment furnace which the West Midlands Gas Board recently has had installed in its Heat Treatment Service establishment located at Adderley Street, Birmingham 9. This establishment was brought into being by the Birmingham Gas Department more than 40 years ago and the plant has been continually brought up to date. The experience of the metallurgical staff of the 'service' is considerable and this is reflected in the quality of the supervision given to all the heat treatment processes carried out at Adderley Street, as well as in the advice given to consumers concerning the use of gas fired plant. This new item of plant, one of the first of its kind to be installed in the Midlands, is an atmosphere controlled batch-type unit, capable of accepting a total load measuring 3 ft. 0 in. wide, 2 ft. 0 in. deep and 1 ft. 6 in. high, and operating up to temperatures of 950°C. The furnace itself comprises two separate chambers, a hot zone, heated by recuperative gas fired nickel-chrome radiant tubes, placed above and below the work chamber; and a water jacketed discharge vestibule. An oil quench tank is incorporated below this vestibule and both the vestibule door and the inner furnace door are power operated.

Controlled atmospheres, suitable for a wide variety of treatments including clean hardening and annealing, gas carburising and carbo-



This Gas-fired furnace of new design for gas carburising and clean heat treatment in controlled atmospheres is a working exhibit at the Board's Heat Treatment Service establishment.

nitriding, are supplied from an endothermic generator forming part of the plant. This generator is fired by town gas but uses propane gas and air for the production of the endothermic gas. Both propane and ammonia enrichment are provided, for use when gas carburising or carbonitriding, and it is possible to vary the 'dew point' of the atmosphere gas in order to provide clean heat treatment of steels over a wide range of specifications.

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stage of the process and the state of the plant at any given time.

In order fully to demonstrate this plant to prospective purchasers it is of course necessary to operate it on a commercial heat treatment shop basis and the Board's Heat Treatment Service is open to receive orders for the gas carburising of such items as crown wheels and pinions, gears, cams and cycle components, clean annealing, hardening, and tempering of special die blocks, machined parts and similar items. All this work would be carried out to the requirements of the client concerned, would be supervised by qualified metallurgists, and when necessary would conform to the dictates of any inspection approval.

Industrialists, metallurgists and plant engineers are invited to inspect any of the items of plant at Adderley Street either with a view to installing similar plant at their own works or for the purpose of assessing the usefulness to their own organization of the commercial heat treatment facilities provided by the 'Service'.

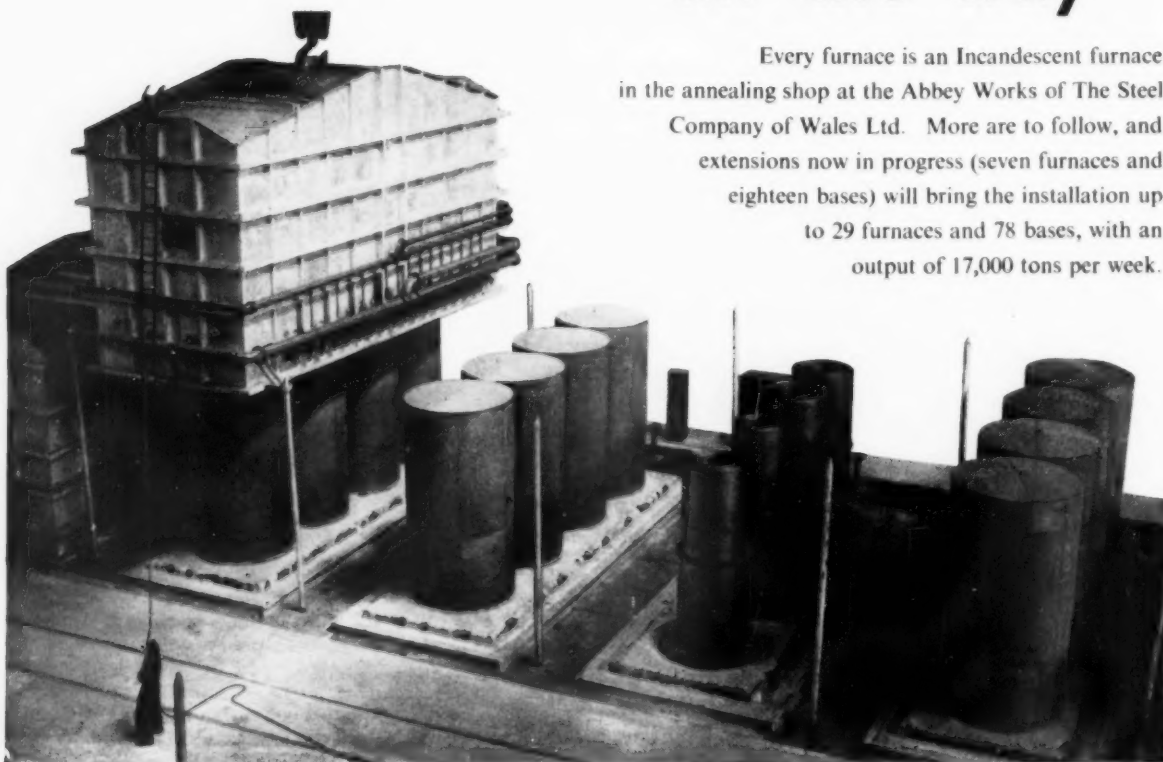
This is an advertisement by the West Midlands Gas Board.



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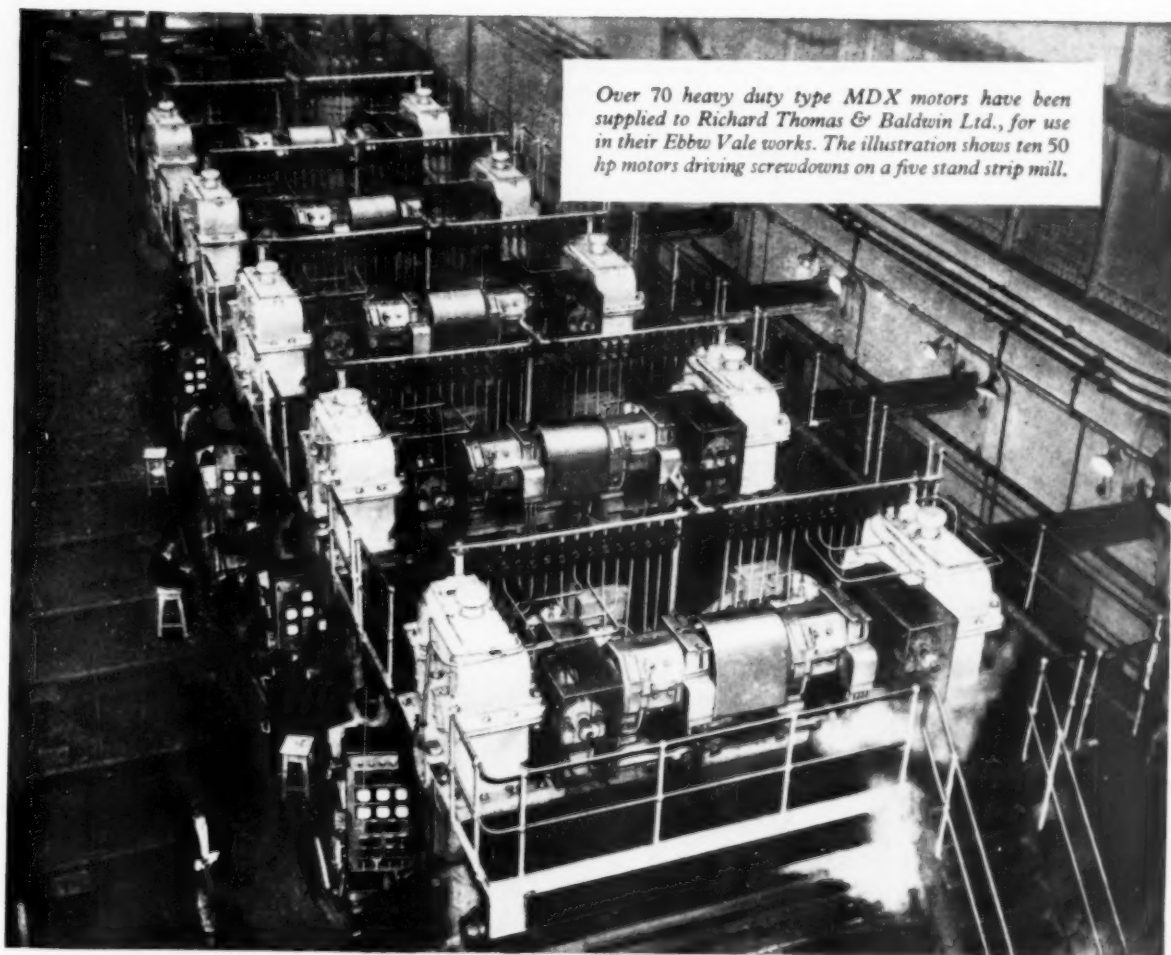
all the way

Every furnace is an Incandescent furnace in the annealing shop at the Abbey Works of The Steel Company of Wales Ltd. More are to follow, and extensions now in progress (seven furnaces and eighteen bases) will bring the installation up to 29 furnaces and 78 bases, with an output of 17,000 tons per week.



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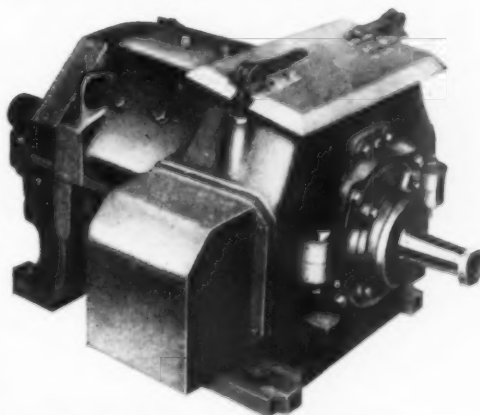
13/10/59



Over 70 heavy duty type MDX motors have been supplied to Richard Thomas & Baldwin Ltd., for use in their Ebbw Vale works. The illustration shows ten 50 hp motors driving screwdowns on a five stand strip mill.

All motors here are type MDX

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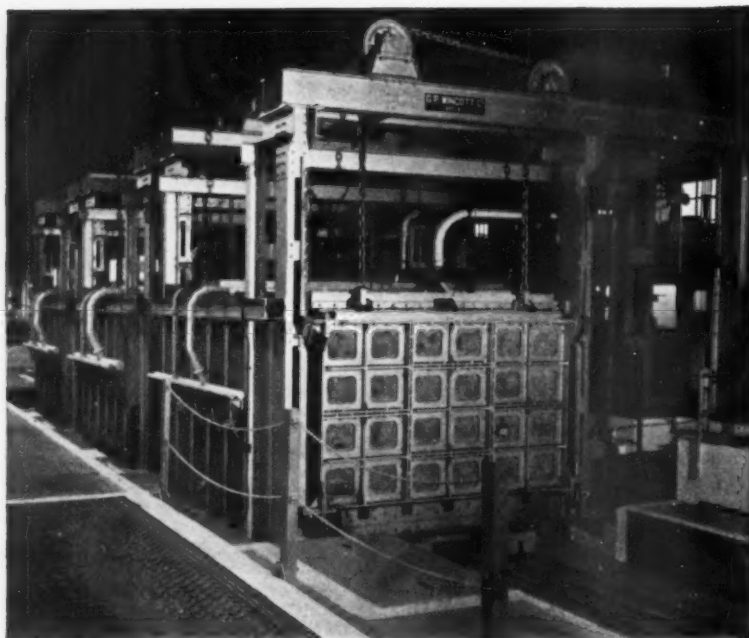
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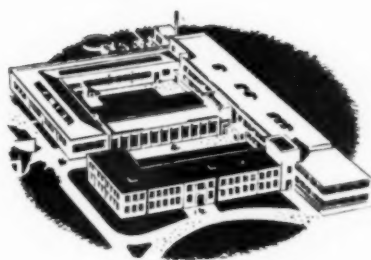
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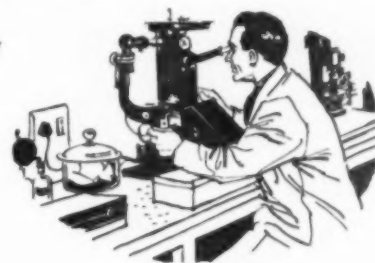
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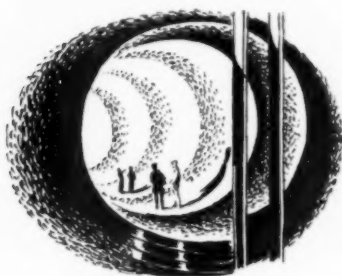
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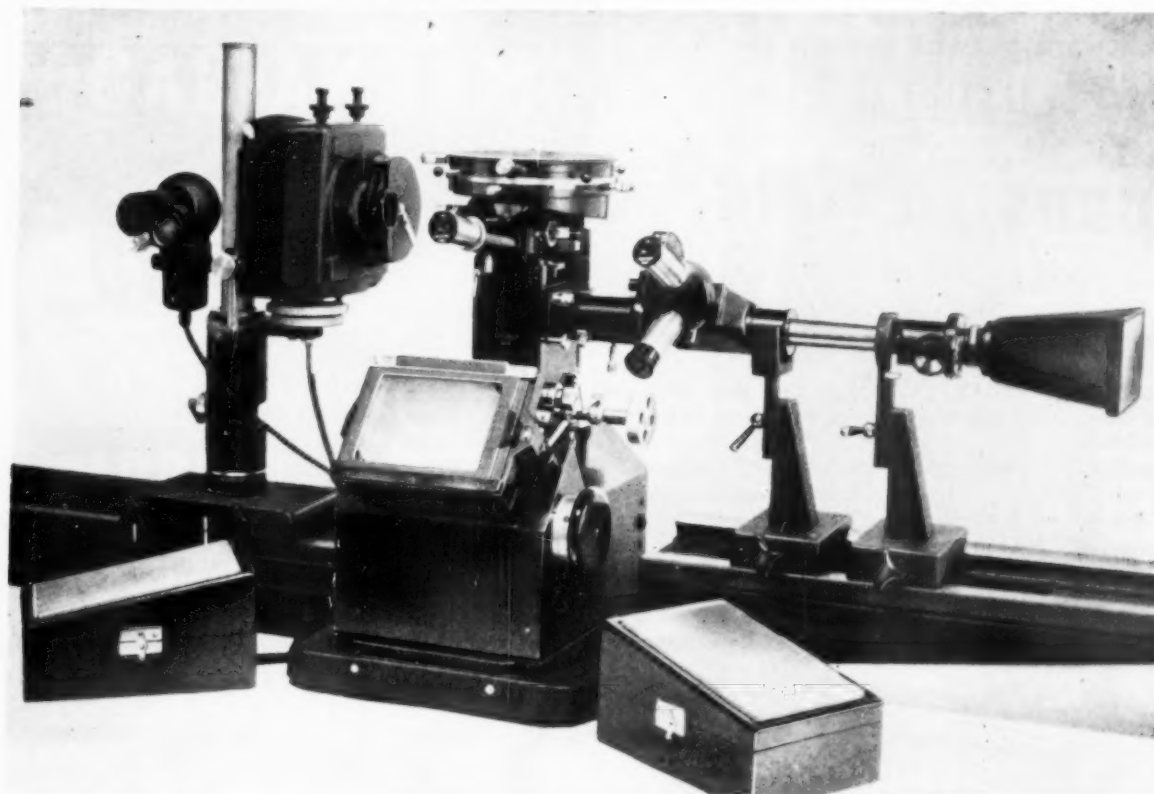
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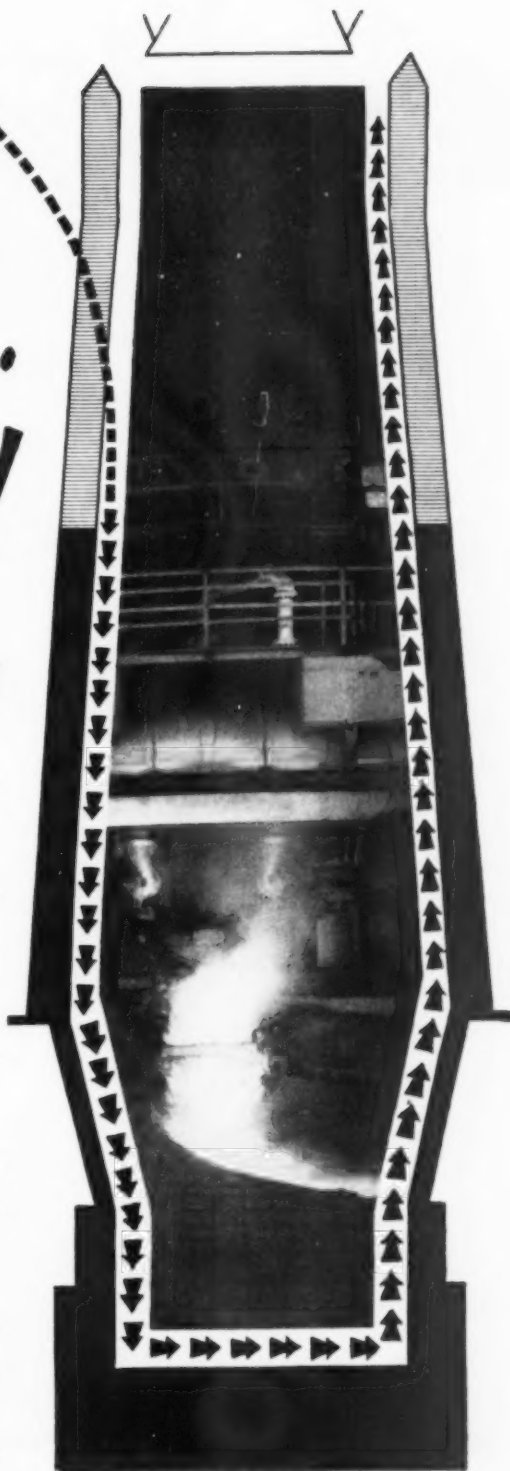
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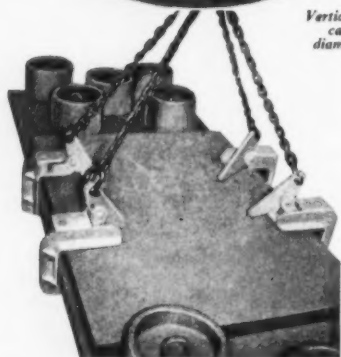


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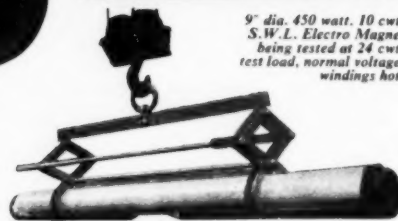
Horizontal cylindrical specified diameter lifting device, also supplied with special clamp faces for handling ground diameters.



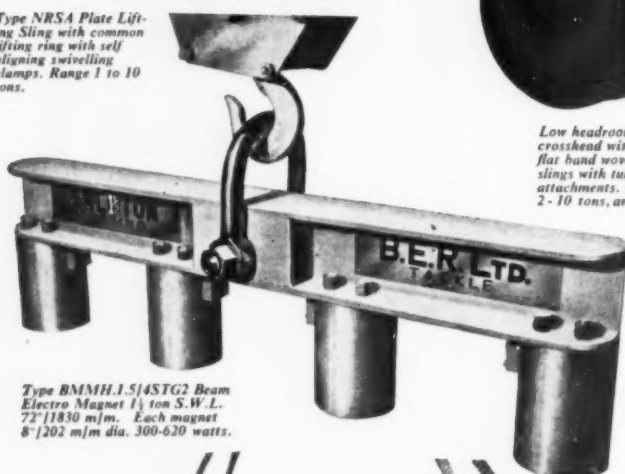
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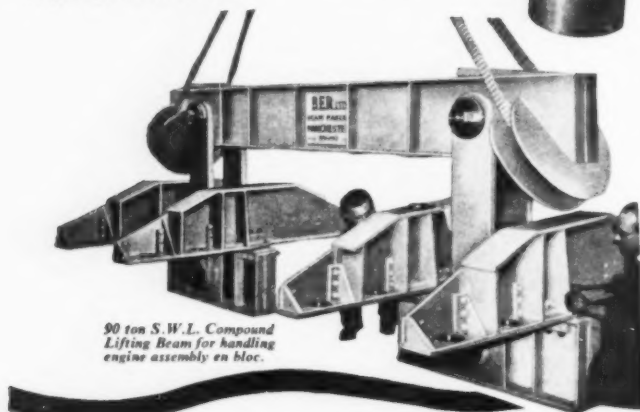


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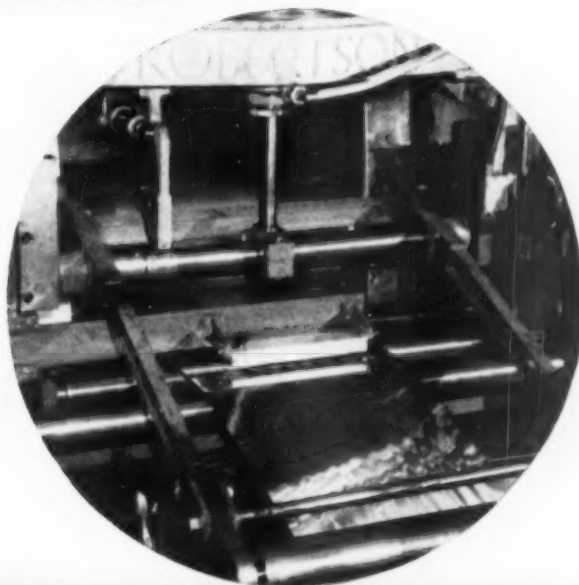
MATERIAL

RAY DETECTOR

Amount of rays passing through material indicates thickness or weight per unit area.

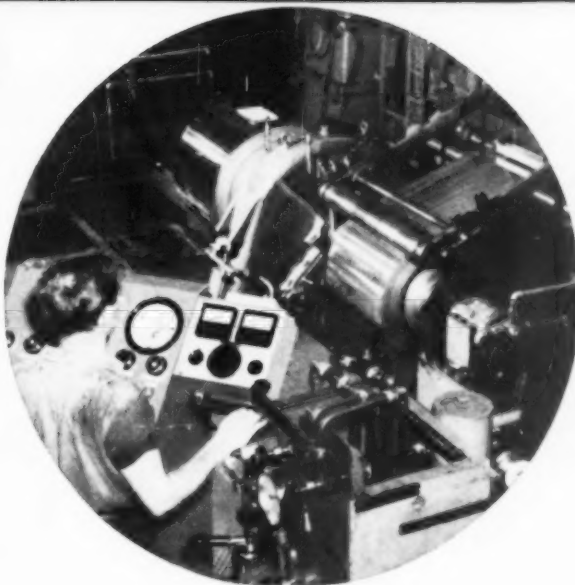
That's the very basic explanation of the Atomat principle. There are three main gauges; the Atomat Cold Strip Gauge, the Hot Strip Gauge and the Beta Gauge.

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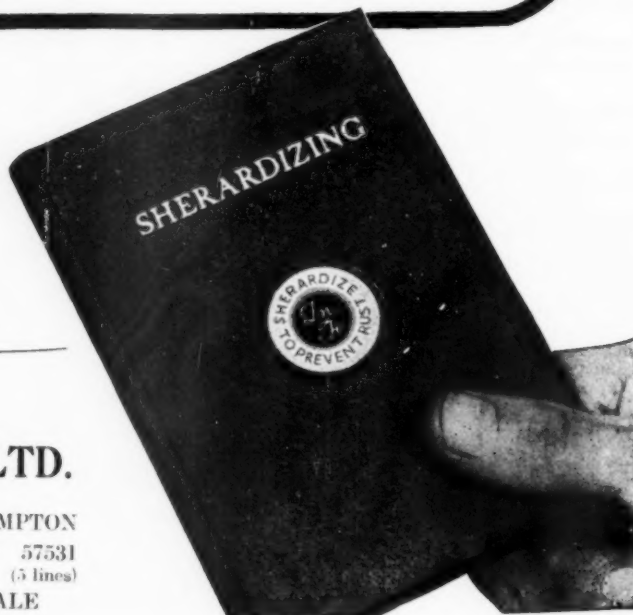
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Lead	below ·01	
Cobalt	·005	·008
Antimony	below ·01	
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THE BRITISH JOURNAL OF METALS
INCORPORATING THE METALLURGICAL ENGINEER

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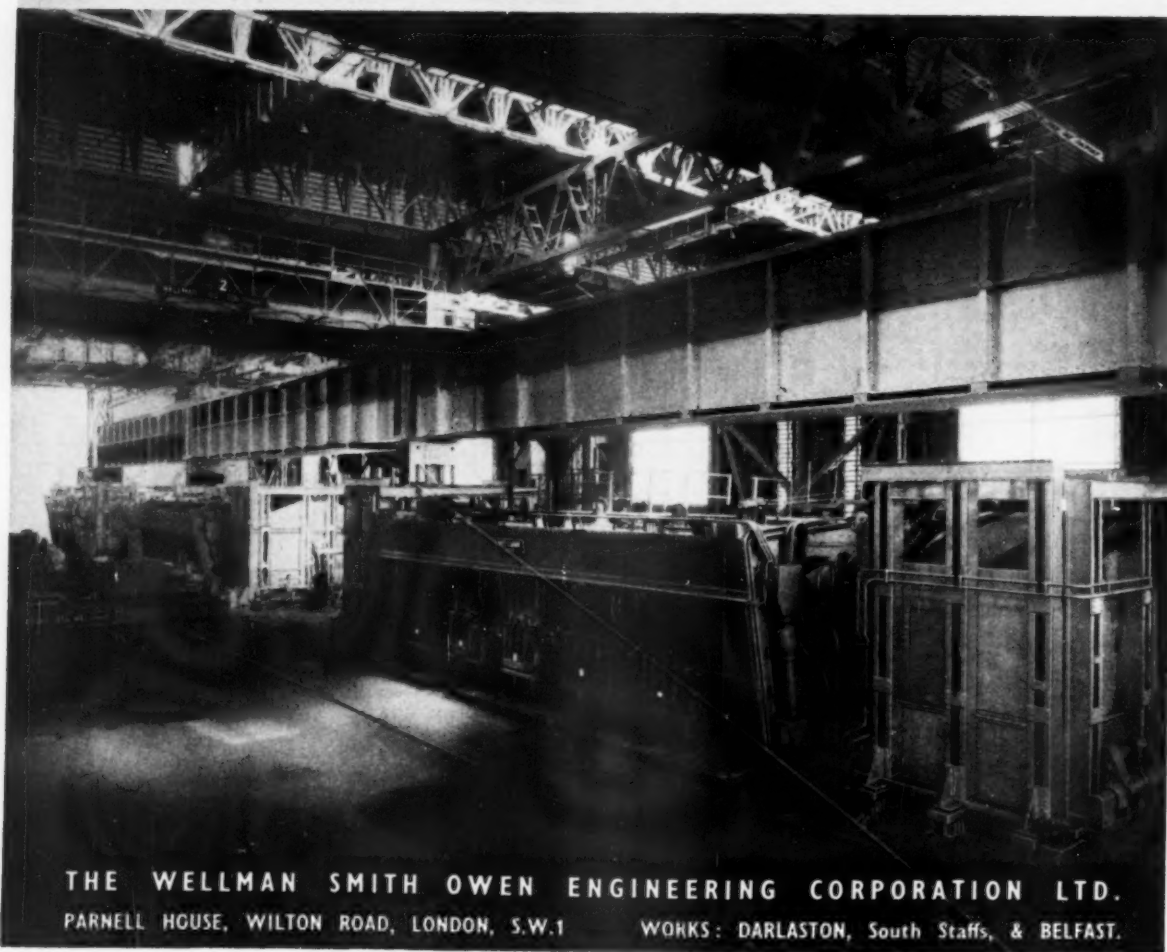
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METALLURGIA

THE BRITISH JOURNAL OF METALS
INCORPORATING THE "METALLURGICAL ENGINEER"

September, 1959

Vol. LX. No. 359

Power from Coal or Atom

THE general energy situation has changed considerably since the announcement by the British Government almost five years ago of a nuclear power plant programme to be completed by 1965. At that time there was a chronic shortage of energy, and it was generally agreed that there would be difficulty in meeting Europe's energy needs from indigenous coal supplies. In the United Kingdom, difficulties arising from the coal shortage in the post-war years had been met, with Government approval, by a switch to other fuels such as oil and—in the steel industry—the less conventional creosote-pitch. Nevertheless, it was felt that, for a variety of reasons, it was undesirable to meet Europe's shortages by large scale importing of oil or of American coal. In these circumstances, the prospect of power from nuclear sources was viewed with a sense of, not merely relief, but rejoicing, and the setting-up of nuclear power stations an obvious move.

What a change has taken place in the last few years. Instead of a shortage of coal in this country, there is today a surplus of such magnitude that the coal mining industry is facing a crisis, and demands are being made by the miners that the Government should take such action in the power production field as to ensure the use of more coal for steam raising, and should encourage the use of coal in other ways. Although the mining industry is one of great importance to the country's economy, it seems a little unfair to expect that industries which have been driven to try other fuels by the shortage—and possibly the cost—of coal should now turn about face and use coal again. Quite apart from the cost involved in alterations to plant, wholesale reversion would probably lead to shortages again. In any case, the governing factor as to which type of fuel is best for power generation is that of cost—or rather the price per kWh of electricity—taking into consideration changes likely to occur in the near future.

When the White Paper on nuclear energy was published in 1955, it was stated that the cost of power from the nuclear stations was expected to be about 0.6d./kWh and that this was about the same as the probable future cost of electricity generated by new coal-fired stations. In a panel discussion at the Sixth International Exhibition on Electricity and Atomic Energy, held in Rome earlier this year, Mr. J. A. Jukes, Economic Adviser to the U.K.A.E.A., discussed the changes which have taken place since those estimates were made, pointing out that two economic factors quite external to the fuel situation have changed, and have had a marked effect on the estimates. Besides a general inflationary rise in the price level of about 10%, the Government's economic policy has increased the cost of money: the rate of interest paid by public utilities has risen from 4% to some 5½%, a change which makes less attractive invest-

ment in nuclear power stations, where capital charges form such a high proportion of production costs. The effect of these changes is to increase the estimated cost of nuclear power by a little more than 0.1d./kWh., and of power from coal by a rather smaller amount. Present estimates suggest that the first nuclear stations will probably produce power at a little over 0.7d./kWh., the most recent stations at or below this figure, and the last station in the programme substantially below it.

The estimated cost of generation in the most up-to-date coal-fired power stations at present being planned ranges from 0.5d./kWh. for stations situated near coalfields to 0.65d./kWh. for similar stations sited away from the coalfields. As far as it is possible to compare the two, the cost of oil generation would be about the same. A rather remarkable change in the economics of coal-fired stations has taken place in the last few years, in that, whereas capital costs of about £70/kW. prevailed four or five years ago, new stations are now expected to be built for a capital cost of £40–50/kW. Gratifying though this reduction is, the proportion of the cost of electricity which is due to capital charges, in the case of coal-fired stations, is so small that there is no great scope for further price reduction due to such technical developments as the use of larger generating sets, etc., even though technological progress continues. The principal cost factor in generation in such stations is the price of coal, varying as it does from 0.35 to 0.5d./kWh., depending on the distance from the coalfield.

In contrast, the fuel cost in the latest design of nuclear stations is 0.17d., which is only about a quarter of the total generating cost. Moreover, it is expected that a substantial fall in the price of uranium will occur in the middle 1960's, because surplus capacity will be available and the mines will be fully amortised. It is estimated that this factor alone will reduce the cost of electricity from existing nuclear stations by about 10% and, if account is also taken of the reductions made possible by lower capital charges, it seems reasonable to hope that the cost of nuclear power will fall below that of conventional power within the next decade.

Whether the nuclear power station will eventually supersede the coal-fired station completely is a matter for conjecture. In the long run it may do so, but for the present, coal-fired stations will continue to be built, and it is predicted that the consumption of coal in the electricity industry will rise substantially in the next few years. In the meantime, of course, progress is to be expected in the development of new types of nuclear stations. The Calder Hall type has several years of development before it, and it is hoped that by 1961 the advanced gas-cooled reactor will become critical. If the latter is successful, the first commercial type may be in operation by 1965. After that the fast breeder reactor under development at Dounreay may come into its own, with its low capital cost and its ability to use the plutonium produced in the Calder Hall type reactors.

Meeting Diary

3rd November

Institute of Metals, Oxford Local Section. "The Theory of Rolling", by PROF. H. FORD. Cadena Cafe, Cornmarket Street, Oxford. 7 p.m.

Institution of Engineers and Shipbuilders in Scotland. "Research on Steel and Aluminium Hatchway Beams", by M. N. PARKER, K. V. TAYLOR, and J. A. ROSS. 39, Elmbank Crescent, Glasgow. 6.30 p.m.

Sheffield Metallurgical Association. "Modern Developments in the Theory of Brittle Fracture", by DR. A. A. WELLS. B.I.S.R.A. Laboratories, Hoyle Street, Sheffield, 3. 7 p.m.

5th November

Institute of Metals, London Local Section. "The Struggle for High Temperatures", by DR. F. D. RICHARDSON. Royal School of Mines, Prince Consort Road, London, S.W.7. 7 p.m.

Liverpool Metallurgical Society. "Ductile Fracture", by PROF. R. W. K. HONEYCOMBE. Library of the Department of Metallurgy, The University of Liverpool, 146, Brownlow Hill, Liverpool, 3. 7 p.m.

Society of Chemical Industry, Corrosion Group. "Corrosion Factors Affecting the Choice of Stainless Steels for Chemical Plant", by H. T. SHIRLEY. Joint Meeting with the Nottingham Section. Gas Showrooms, Shakespeare Street, Nottingham. 7.30 p.m.

10th November

Institute of Metals, South Wales Local Section. "Economics of Rolling Mill Layouts", by W. F. CARTWRIGHT. Metallurgy Department, University College, Singleton Park, Swansea. at 6.40 p.m.

Sheffield Metallurgical Association. "Silicon Carbide and other Super Refractories—Recent Developments", by C. PRESSWOOD. B.I.S.R.A. Laboratories, Hoyle Street, Sheffield, 3. 7.30 p.m.

11th November

Institute of Metals. Conversazione (informal) in connection with Electron-Microscope Symposium. 17, Belgrave Square, London, S.W. 1. 7.30 p.m. Tickets 6s. each.

Liverpool Metallurgical Society. "Welding in Warship Construction", by W. R. SEWARD. Joint Meeting with the Institute of Welding, Liverpool Branch. Picton Library, Liverpool. 7.30 p.m.

Manchester Metallurgical Society. "Bearing Metals", by P. G. FORRESTER. Manchester Room of the Central Library, St. Peter's Square, Manchester. 6.30 p.m.

12th November

East Midlands Metallurgical Society. "Metals in Storage Batteries", by C. J. BUSHROD. The Central Electricity Authority Showrooms, Arkwright Street, Nottingham. 7.30 p.m.

Institute of Metals. Symposium on "The Application of Thin-Film Techniques to the Electron Microscopic Examination of Metals", arranged by the METAL PHYSICS COMMITTEE, Royal Institution, Albermarle Street, London, W.1. 9.30 a.m., and 2.15 p.m.

Institute of Metals, Birmingham Local Section. "Recent Corrosion Studies on Titanium", by J. B. COTTON. College of Technology, Gosta Green, Birmingham. 6.30 p.m.

Leeds Metallurgical Society. "Aluminium Casting Alloys and Foundry Practice", by W. L. BOLTON. Metropole Hotel, Leeds. 6.30 p.m. Light refreshments available from 6 p.m.

14th November

Non-Destructive Testing Society of Great Britain, Birmingham Branch. "The Logical Development of an N.D.T. Technique for a Steel Casting", by S. JURY. Engineering Centre, Stephenson Place, Birmingham. 10 a.m.

17th November

Sheffield Metallurgical Association. "Modern Developments in Ultrasonic Inspection Methods", by J. F. HINSLEY. B.I.S.R.A. Laboratories, Hoyle Street, Sheffield, 3. 7 p.m.

18th November

Southampton Metallurgical Society. Joint Meeting with the **Institute of British Foundrymen.** "Properties of Copper Base Alloy Castings", by F. HUDSON. Southampton Technical College. 7.30 p.m.

19th November

Institute of Metals, Sheffield Local Section. "Primitive Metallurgy", by PROF. F. C. THOMPSON. Applied Science Building of the University, St. Georges' Square, Sheffield. 7.30 p.m.

Institution of Plant Engineers, Merseyside and North Wales Branch. "Developments in Welding Practice", by DR. R. WECK. Donnan Laboratories, Liverpool University. 7 p.m.

20th November

West of Scotland Iron and Steel Institute. "Metallurgical Aspects of Flat Rolled Products", by A. J. K. HONEYMAN. 39, Elmbank Crescent, Glasgow. 6.45 p.m.

24th November

Institution of Plant Engineers, South Wales Branch. "The Engineering Applications of Electro-Deposited Metals", by J. W. OSWALD. South Wales Engineers' Institute, Park Place, Cardiff. 7.30 p.m.

Sheffield Metallurgical Association. Symposium on the Determination of Carbon in Steel. "Gravimetric Methods", by E. A. DICKINSON. "Low Pressure Method", by R. STATHAM. "Conductimetric Method", by W. R. NALL. B.I.S.R.A. Laboratories, Hoyle Street, Sheffield, 3. 7 p.m.

25th-27th November

Institute of Petroleum and Society of Chemical Industry (Corrosion and Chemical Engineering Groups). Symposium on "Corrosion Problems of the Petroleum Industry." Federation of British Industries, Tothill Street, London, S.W.1.

Nov. 25th. Eve of Symposium Reception, Washington Hotel, Curzon Street, London, W.1. 6 p.m.

Nov. 26th 9.30 a.m. to 12.30 p.m. "Corrosion of Production Equipment and Gathering Lines and its Prevention," by G. A. LEE and G. A. HAINES; "Corrosion of Marine Structures and its Prevention," by G. T. COLEGATE.

2.30 p.m. to 5.30 p.m. "Corrosion of Tanker Hulls and its Prevention," by J. G. ROBINSON and K. FLEMING; "Recent Developments in the Protection of Pipelines and Storage Tanks," by H. B. FOOTNER and P. W. HESELGRAVE.

Nov. 27th 9.30 a.m. to 12.30 p.m. "Corrosion Prevention in Salt Water Cooling Systems" (two papers), by P. T. GILBERT and E. D. DOLAN.

2.30 p.m. to 5.30 p.m. "Non-Destructive Testing," by L. WILKINSON and P. S. COTTEN; "Developments in Corrosion-Resistant Materials," (a) "Metals," by G. L. SWALES; (b) "Non-Metallic Materials," by I. H. THOMAS and T. S. McROBERTS.

25th November

Institution of Mechanical Engineers. Thomas Hawksley Lecture. "The Effect of Nuclear Radiation on Engineering Materials," by PROF. A. H. COTTRELL. 1, Birdcage Walk Westminster, London, S.W.1. 6 p.m.

Manchester Metallurgical Society. "Engineering Aspects of Civil Nuclear Power Stations", by B. D. WILLSON. Manchester Room of the Central Library, Manchester. 6.30 p.m.

26th November

Institute of Metals, Birmingham Local Section. "A Metallurgical Cocktail," by M. A. WHEELER. College of Technology, Gosta Green, Birmingham. 6.30 p.m.

Institution of Mechanical Engineers. Applied Mechanics Group Discussion. "The Application of Creep Results to Engineering Design." 1, Birdcage Walk, Westminster, London, S.W.1. 6 p.m.

Institution of Plant Engineers, Sheffield and District Branch. "Some Modern Steel for the Engineer", by E. JOHNSON. Grand Hotel, Sheffield. 7.30 p.m.

Southampton Metallurgical Society. "Hydrogen in Steel", by J. HEWITT. Engineering Block, Southampton University. 7.15 p.m.

27th November

Institution of Plant Engineers, Birmingham Branch. "Instrumentation in Industry with Particular Reference to Control of Temperature, Pressure Flow, Weight, etc.", by L. F. COHEN. Imperial Hotel, Temple Street, Birmingham. 7.30 p.m.

The Influence of Chromium Diffusion upon the Thermal Oxidation Properties of Steel and of Nickel- and Cobalt-Base Alloys

By R. L. Samuel, Ing. Chim., A.I.M.,* and T. P. Hoar, M.A., B.Sc., Ph.D., F.R.I.C., F.I.M.†

The enrichment with chromium of the surfaces of steels, and of nickel- and cobalt-base alloys, results in improved oxidation resistance. Even with intrinsically oxidation-resistant materials, the treatment, besides lowering the rate of oxidation, may also alter the nature of the oxidation, which may, in untreated material, proceed in an undesirable manner, such as by intergranular penetration.

THE addition of substantial amounts of chromium to iron, nickel or cobalt considerably increases their resistance to thermal oxidation. Examples are the stainless irons and steels with 12-25% of chromium, nickel alloyed with 20-40% of chromium, and cobalt alloyed with 25-35% of chromium. Pure iron, nickel and cobalt (and iron, nickel and cobalt containing small amounts of chromium) are relatively readily oxidized.

Since thermal oxidation is a surface reaction, the enrichment with chromium of the surfaces of iron-, nickel- and cobalt-base alloys is a logical method of improving their resistance to thermal oxidation. Chromium diffusion or "chromizing"^{1,4} is an effective way of achieving this.

General Characteristics of Chromium Diffusion

Most methods of chromium diffusion rely upon the reaction, at temperatures of 850-1,200° C., of a chromium halide upon the surface of the metal to be chromized. Processes may be gaseous (retorts containing the metal to be chromized, over which chromium-bearing gas is passed), or semi-gaseous (boxes or retorts containing also chromium or a high chromium alloy and a controlled atmosphere).^{1,4}

The general characteristics of the diffusion coatings are

* Formerly Technical Director, Diffusion Alloys, Ltd., London; now with Metal Surfaces Research Laboratories, London.

† Department of Metallurgy, University of Cambridge.

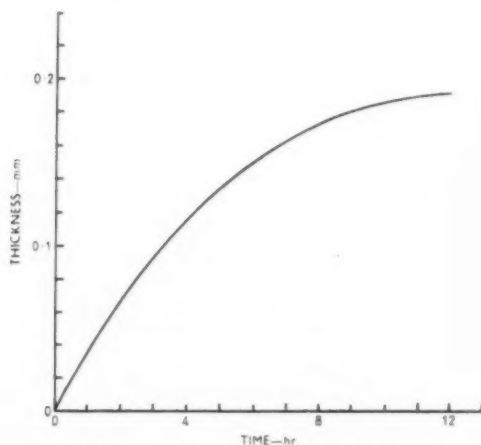


Fig. 1.—Thickness of coating obtained by chromium diffusion at 1,100° C. for various times of treatment.

shown in Figs. 1-4, which relate to the process utilizing chromium iodide as the gaseous chromium compound for the treatment of dead-mild steel (0.1% C); the conditions of processing ensured a constant "saturation" supply of chromium at the surface. The full line in Fig. 4 (concentration of chromium in relation to distance from surface) represents the condition of a specimen after treatment. When such a specimen is reheated at temperatures within or above the processing range, there is a further inward diffusion of chromium dependent upon time and temperature; this is slower than diffusion with a saturation supply of chromium at the surface. Such "secondary" diffusion, represented by the dotted line in Fig. 4, and illustrated in Figs. 5 and 6, causes a lowering in the chromium content at the surface and a migration inwards of the coating boundary, which, in the case of a low carbon steel, occurs at a concentration of approximately 12% chromium. The effect may be important in the thermal oxidation of chromized materials, because it may attain appreciable proportions at the temperatures of utilization of the treated parts.

In all heat-resisting materials, the protection of the surface depends upon the formation of a stable oxide film. The degree of protection depends upon the composition, continuity and adhesion of the film. The diffusion rates of metal ions, oxide ions and oxygen through the film must be as low as possible; Kubaschewski and Hopkins⁵ have summarized the general theory.

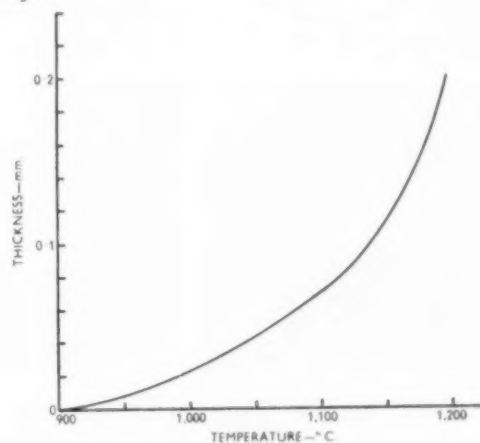


Fig. 2.—Thickness of coating obtained by a 4.5 hour chromium diffusion treatment at various temperatures.

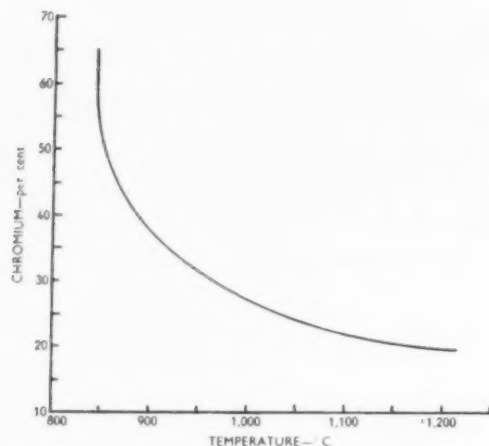


Fig. 3.—Average chromium content of the coating formed on low carbon steels at various temperatures.

In alloys of iron, nickel or cobalt containing considerable amounts of chromium, preferential oxidation leads to the formation of Cr_2O_3 or of spinel-type oxides such as $\text{Cr}_2\text{O}_3\cdot\text{FeO}$, $\text{Cr}_2\text{O}_3\cdot\text{NiO}$ or $\text{Cr}_2\text{O}_3\cdot\text{CoO}$. From the point of view of oxidation-resistance, spinel formation is undesirable, according to Preece and Lucas,⁶ although Hauffe⁷ considers, on the contrary, that it is helpful. There is a higher proportion of chromium in the oxide film than in the surface alloy, of which the chromium content falls as the oxide film grows. Thus the oxidation properties of a diffusion coating depend upon:—

- the intrinsic rate of oxidation corresponding to the surface (actually coating) composition,
- the secondary diffusion effect, which lowers the chromium content of the coating quite apart from any change due to its oxidation.

Factor (a), which is similar to that for plain alloys, is the only important factor in the moderate range of temperatures, say up to 900°C ., for a low carbon steel. Factor (b) becomes increasingly important as the temperature is raised above 900°C ., when the interdiffusion rate of chromium and the basis material reaches serious levels, and thus leads to an increase in the overall rate of oxidation in the higher range of temperatures. The problem is, therefore, a complex one depending upon, among other things, the nature of the basis material.

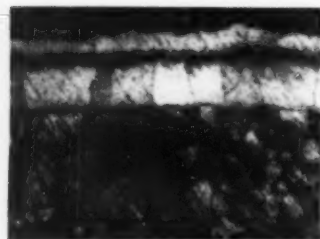


Fig. 5. (left)—Structure of chromium-diffused mild steel. Etched Marble's reagent. $\times 100$

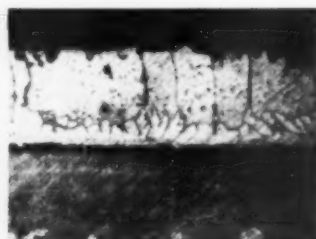


Fig. 6 (right)—Structure of the same specimen as Fig. 5, after reheating 1 hour at $1,200^\circ\text{C}$. $\times 100$

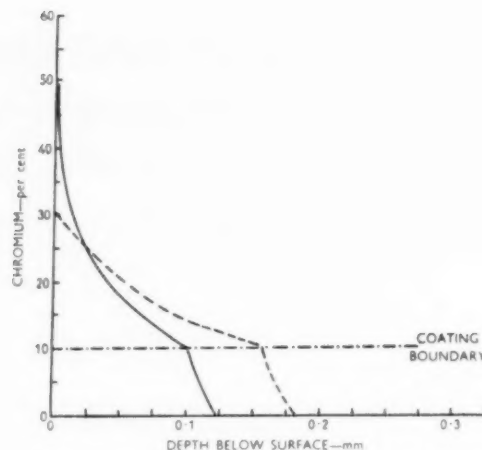


Fig. 4.—Chromium concentration as a function of depth below the surface of chromium-diffused mild steel. The broken curve illustrates the effect of re-diffusion.

Method of Testing

In the experiments here reported, oxidation rate was measured by the increase of weight due to oxygen uptake on cylindrical specimens approximately 1 cm. in dia. \times 1 cm. high. These were suspended by means of platinum wires in thermostatically controlled vertical tubular electric furnaces. A slight air draught of constant intensity was maintained by means of openings at both ends of the heated tube. Several furnaces were mounted side by side on a rigid metal frame fitted with rails on top, along which an analytical balance could be moved to any desired position.

Weighings were taken on the hot specimens at 24-hour intervals. A period of slow cooling, followed by reheating in 2 hours to temperature, was allowed after every 100 hours of continuous heating. After completion of the

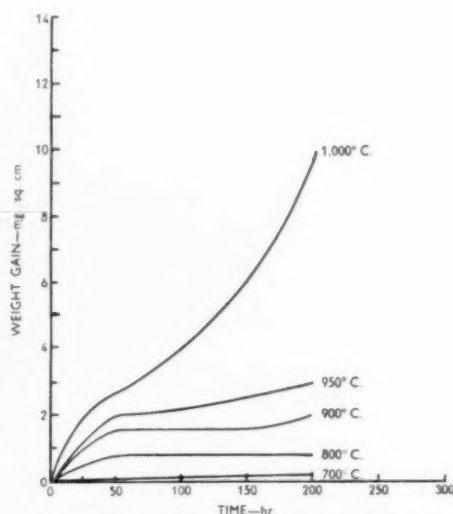


Fig. 7.—Air oxidation of chromium-diffused mild steel (0.1% C) at various temperatures.

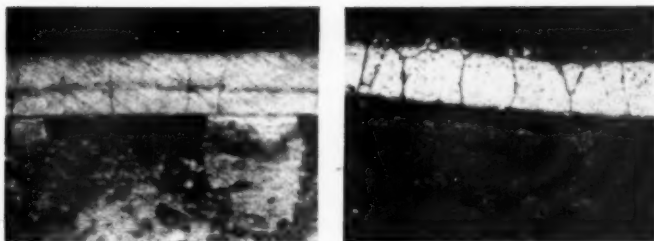


Fig. 8 (left)—Structure of chromium-diffused mild steel before oxidation.

Fig. 9 (right)—Structure of the same specimen as Fig. 8, after air oxidation at 1,000° C. for 36 hours.

test, each specimen was examined visually for type of oxidation (scaling, blistering, etc.). Micro-examinations were carried out on sections in order to reveal further the nature of the oxidation (e.g. sub-scale formation) and any effects upon the microstructure.

Experimental Results

PLAIN CARBON STEELS

Dead-Mild Steels.—The weight increases of a chromized 0.1% carbon steel at temperatures varying from 700–1,000° C. are shown graphically in Fig. 7.⁸ The curves corresponding to temperatures of 700, 800 and 900° C. show a slow rate of oxygen uptake, becoming almost negligible, behaviour similar to that of a conventional heat-resisting alloy. In contrast, the curves for 950 and 1,000° C. show a sharp upward trend, after an initial slow rate. This is caused by the superimposition of the effect of secondary diffusion upon the oxidation rate of the original surface.

The oxide film was usually continuous, with a rate of growth broadly equal at all parts of the surface, as shown in Figs. 8 and 9.

Medium and High Carbon Steels.—The effect of carbon content of the basis steel upon its rate of thermal oxidation after chromizing is shown in Fig. 10, which refers to tests at 950° C., a temperature sufficiently high to lead to marked differences in oxidation behaviour. Figs. 11–14 illustrate features of the oxidation mechanism.

There are two salient points:

(a) *The Chemical Composition of the Steel.* Carbon in the steel retards diffusion of chromium in depth and, as a result, coatings obtained on high carbon steels are thin but of high chromium content. Part of this chromium is present in the form of carbides. The rate of oxidation is consistent with a chromium content of the order of 50% or more, and secondary diffusion is slow.

(b) *The Structure underneath the Coating.* At 950° C., all the steels giving the results shown in Fig. 10 are in the single-phase austenitic condition. On cooling, the 0.2% carbon steel transforms to a nearly completely ferritic structure, and the 1% carbon steel transforms to a completely pearlitic structure. The 0.4% carbon steel, however, develops about equal amounts of ferrite and pearlite, and this structure probably introduces stresses in the coating during cooling, causing fractures that expose

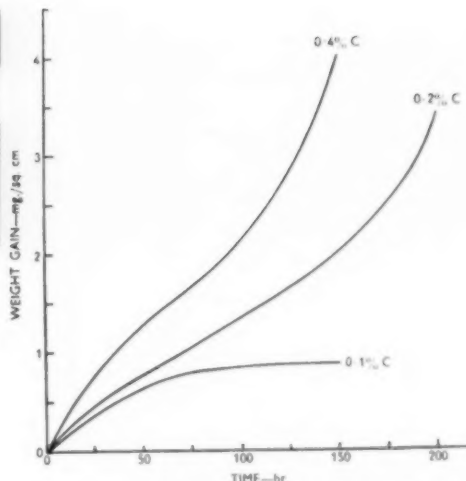


Fig. 10.—Air oxidation of chromium-diffused carbon steels (0.2, 0.4 and 1.0% C.) at 950° C.

the core at places and considerably increase the net rate of subsequent oxidation.

Fig. 12 shows a typical “mushroom” of oxide that has developed at a crack in the coating, clearly illustrating iron diffusion outwards through the growing oxide film, as first demonstrated by Pfeil.⁹ The coating itself is seen to be scarcely oxidized. Evidently, the microstructure of the metal immediately underneath the coating may be a determining factor in the oxidation resistance of the chromized processed material.

Fig. 15, after Sully, Brandes and Brown¹⁰ shows the comparative behaviour of two types of aluminium-

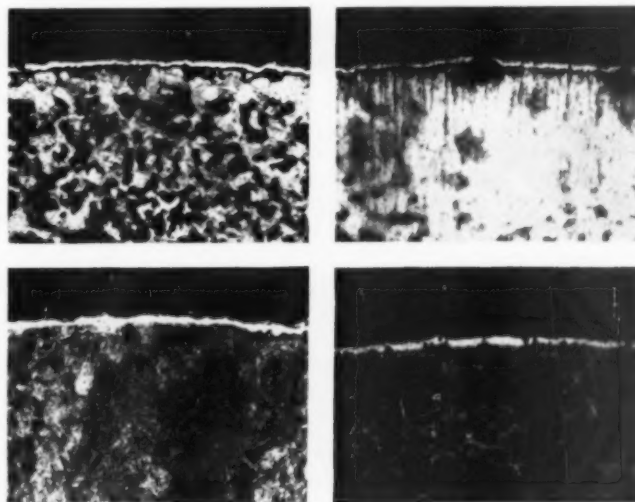


Fig. 11 (top left)—Structure of chromium-diffused 0.4% C steel. Etched Nital.

Fig. 12 (top right)—Structure of the same specimen as Fig. 11, after air oxidation at 950° C. for 138 hours.

Fig. 13 (bottom left)—Structure of chromium-diffused 1% C steel. Etched Nital.

Fig. 14 (bottom right)—Structure of the same specimen as Fig. 13, air oxidation at 950° C. for 307 hours.

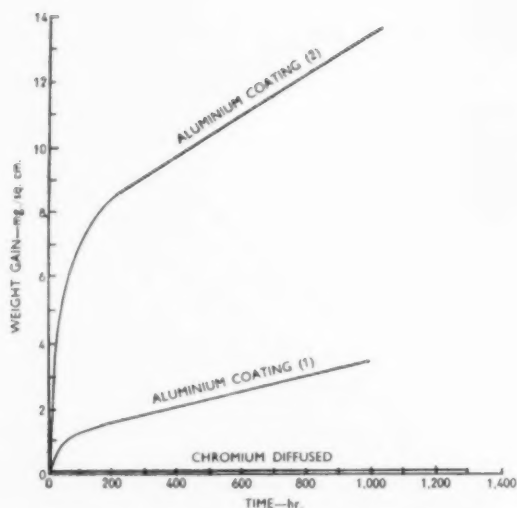


Fig. 15.—Comparison between chromium diffusion and aluminum coatings on a 1% C steel at 700° C., continuous heating (after Sully, Brandes and Brown¹⁰).

coated steel and of a chromized 0.1% carbon steel at 700° C. in a 1,000 hour continuous heating test, together with a curve for untreated steel. The superior oxidation resistance of the chromized material is very marked.

LOW-ALLOY STEELS

The general behaviour of low alloy steels is similar to that of plain carbon steels. Thus, Fig. 16 gives typical results for the following steels.

1% Cr-Mo-V Steel (0.10% C)

This steel develops a homogeneous microstructure underneath the coating on cooling. The rate of oxida-

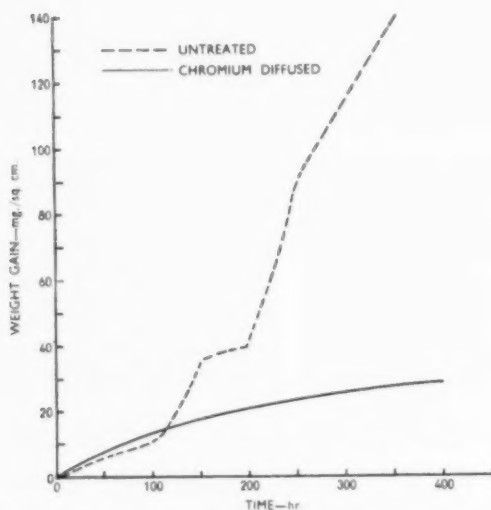


Fig. 17.—Air oxidation of creep-resisting austenitic steel at 950° C.

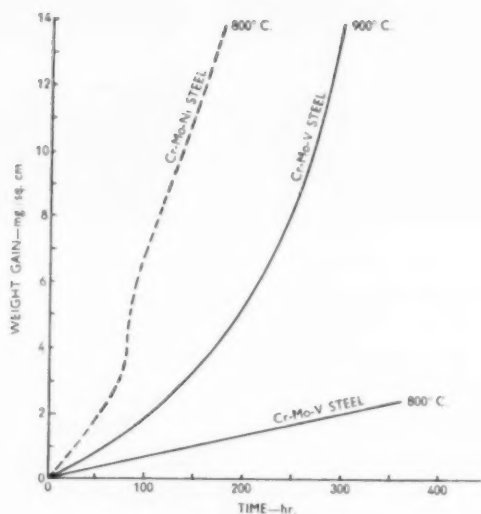


Fig. 16.—Air oxidation of chromium-diffused low alloy steels at 800° C. and 900° C.

tion is low at 800° C.; at 900° C. the effect of secondary diffusion is noticeable.

1% Cr-Ni-Mo Steel (0.07% C)

This steel develops a non-homogeneous microstructure underneath the coating on cooling, and tends to fail in the same way as a 0.4% carbon steel. Even at 800° C. the net rate of oxidation is considerable.

HIGH-ALLOY STEELS

A creep-resisting austenitic steel that has found considerable use in gas and aircraft turbines has nominal composition: C, 0.4%; Mn, 0.8%; Si, 1.0%; Ni, 13%; Cr, 13%; Co, 10%; Mo, 2%; Nb, 3.0%; W, 2.5%; Fe, balance. This steel possesses a measure of intrinsic oxidation resistance. The effect of chromium diffusion is shown in Fig. 17; after 400 hours at 950° C.

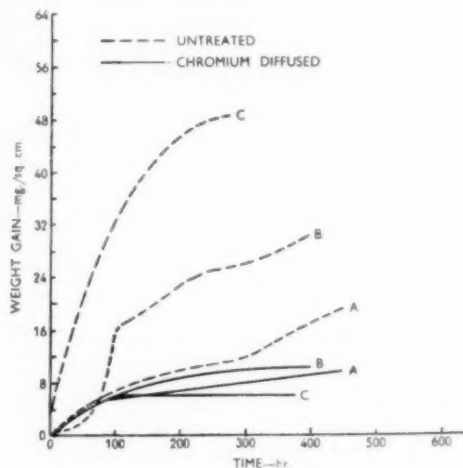


Fig. 18.—Air oxidation of nickel-base alloys A, B and C.

the amount of oxidation on the chromized sample is approximately one-sixth that of the untreated material. The secondary diffusion effect is insignificant.

NICKEL-BASE ALLOYS

Nickel and nickel alloys can be chromized in the same manner as irons and steels. The general properties of the coatings have been described elsewhere,¹¹ and we shall limit our discussion to three alloys, *A*, *B* and *C*, that are widely used for high temperature applications. These have typical compositions:—

	<i>A</i>	<i>B</i>	<i>C</i>
Carbon ..	0.04%	0.06%	0.09%
Titanium ..	1.8-2.7%	2.25%	2.84%
Chromium ..	18-21%	18-21%	19.9%
Aluminium ..	0.5-1.8%	1.24%	1.70%
Silicon ..	1% max.	1.5% max.	1.0% max.
Manganese ..	1% max.	1% max.	1% max.
Iron ..	5% max.	5% max.	5% max.
Cobalt ..	2% max.	15.2%	16.5%
Copper ..	—	—	0.5% max.
Nickel ..	Balance	Balance	Balance

These alloys, in addition to having considerable strength at high temperatures, are classified as oxidation-resistant.

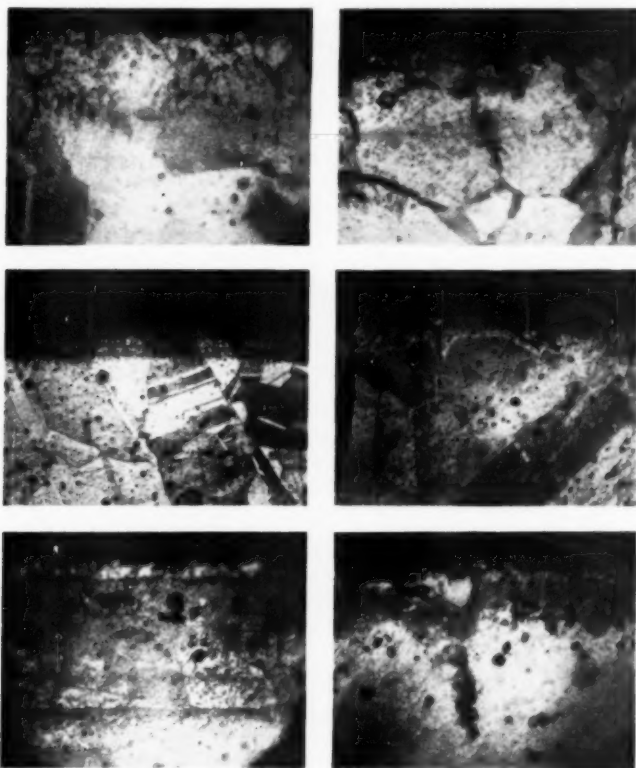


Fig. 20 (top left)—Structure of chromium-diffused nickel-base alloy *A* after air oxidation at 950° C. for 450 hours. $\times 190$
 Fig. 21 (top right)—Structure of untreated nickel-base alloy *A* after air oxidation at 950° C. for 450 hours. $\times 190$
 Fig. 22 (centre left)—Structure of chromium-diffused nickel-base alloy after air oxidation at 950° C. for 380 hours. $\times 50$
 Fig. 23 (centre right)—Structure of untreated nickel-base alloy *C* after air oxidation at 950° C. for 380 hours. $\times 50$
 Fig. 24 (bottom left)—Structure of the same specimen as Fig. 22, showing details of oxide inclusions. $\times 190$
 Fig. 25 (bottom right)—Structure of the same specimen as Fig. 23, showing details of oxide inclusions. $\times 190$

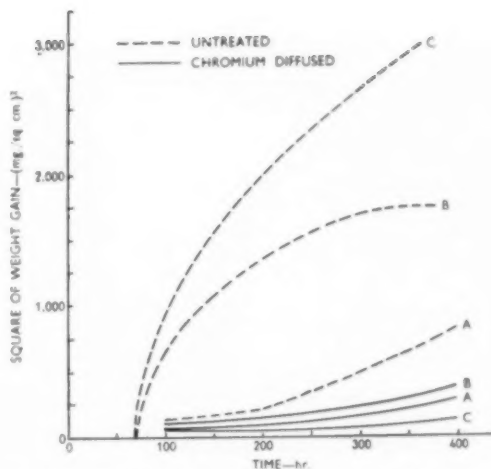


Fig. 19.—Same data as Fig. 18, "parabolic" plot.

However, they may develop intergranular oxidation when subjected to prolonged exposure to oxidizing atmospheres at temperatures of heat-treatment or of utilization. The mechanism of this oxidation may well be chromium-, aluminium- and titanium-oxide precipitation at grain boundaries and near the surface.^{12, 13}

By diffusing chromium into the surface of these alloys, it is possible to alter their oxidation characteristics. Fig. 18 illustrates the comparative rates of oxidation at 950° C. of alloys *A*, *B* and *C* in the chromized and untreated conditions, when tested as described above. It can be seen that all the chromized materials show a much reduced rate of oxidation and fall within the same range, irrespective of the basis material. Evidently the high surface chromium content largely cancels out the effect of differences of composition in the underlying alloy. In Fig. 19 (weight increase, unit area)² is plotted as ordinate. All the points relating to chromized materials fall on a group of nearly coincident straight lines indicating nearly "parabolic" oxidation. In marked contrast, the untreated materials show higher rates of oxidation not following a simple law, probably because of a complex mechanism of oxidation.

The differences in the nature of the oxidation are illustrated in Figs. 20-24. The following general features are apparent:

- The extent of the zone affected by oxidation is reduced in the treated specimens.
- The chromized specimens exhibit oxide inclusions which are generally small and distributed throughout the surface alloy. In the untreated specimens, massive oxide inclusions, preferentially distributed along grain boundaries, are formed.

Clearly, through the alteration of the type of oxidation, chromium-enrichment at the surface may have a profound indirect influence upon the physical properties of this group of alloys at elevated temperatures, apart from its direct influence upon the rate of oxidation.

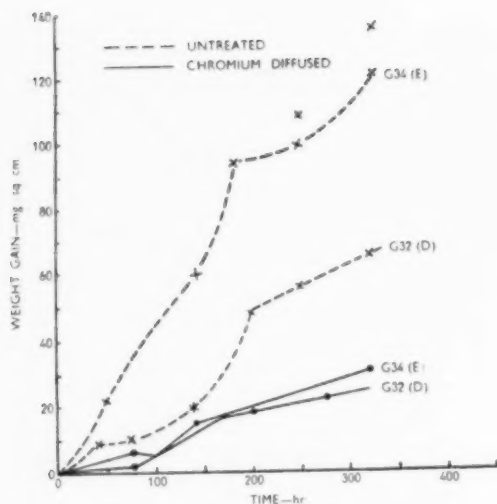


Fig. 26. Air oxidation of cobalt-base alloys D and E.

COBALT-BASE ALLOYS

Two alloys, *D* and *E*, of the high temperature, creep-resisting type were tested. The compositions were:

	<i>D</i>	<i>E</i>
	(casting variety of <i>D</i>)	
Carbon	0.3%	0.5-1%
Manganese	0.8%	0.8%
Silicon	0.3%	0.5%
Nickel	12.0%	12.5%
Chromium	19.0%	19%
Cobalt	45%	45%
Molybdenum	2.0%	2.8%
Niobium	1.2%	1.3%
Vanadium	2.8%	2.8%
Iron	Balance	Balance

Their oxidation rates at 950° C., in the chromized and untreated conditions, are shown in Figs. 26 and 27. The qualitative behaviour of these alloys is similar to that of the nickel-base alloys, and the remarks made in the preceding section broadly apply. However, the absolute rates of oxidation are appreciably higher, although in the case of the chromized specimens they appear to follow approximately the simple parabolic equation.

It may be noted that Preece and Lucas⁶ found, for the simple binary cobalt-chromium alloys, high oxidation resistance only with a chromium content greater than 25%; below this, the cobalt chromite spinel forms in the oxide film, which is much less protective. It is thus not surprising that the increase of surface chromium content in alloys *D* and *E* (19% Cr) by chromizing improves their oxidation resistance.

Summarized Conclusions

It seems clear that surface chromium diffusion can provide better oxidation resistance for a wide range of materials. Such treatment does not necessarily alter the bulk properties of an alloy, which may well have been formulated with properties other than oxidation resistance primarily in mind. Plain carbon and low alloy steels, materials that are not inherently oxidation-resistant, can be very considerably protected by a surface layer of chromium-rich alloy, and thereby brought to the level of a high alloy steel. When intrinsically oxidation-resistant materials, such as high alloy steels and nickel-

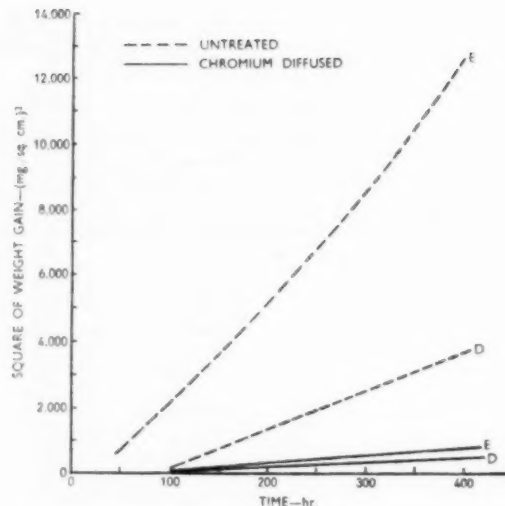


Fig. 27.—Same data as Fig. 26, "parabolic" plot.

or cobalt-base materials, are chromized, the treatment often has the useful dual effect of further lowering the rate of oxidation and of altering the nature of the oxidation, which may, in the untreated material, proceed in an undesirable manner such as by intergranular penetration.

We have represented each group of materials by typical examples; this paper does not by any means cover the whole range of available alloys and treatments. Particularly when dealing with complex alloys, it is difficult and indeed dangerous to generalize, and each case must be studied separately. However, the results described above apply broadly (though not quantitatively) to many other alloys in the respective groups.

Chromium-diffusion can also produce a marked reduction in the oxidation rates of many alloys in the presence of the combustion products of fuels, such as CO, CO₂, SO₂, H₂S, etc. This is a wide subject that cannot be considered within the scope of simple thermal oxidation.

Considerable industrial use is already being made of chromium-diffused coatings,¹⁴ and a more general knowledge of their properties will no doubt bring to light many other practical applications.

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THE address of the Bristol Branch Office of The English Electric, Co., Ltd., is now Equity and Law Building, 36/38 Baldwin Street, Bristol, 1 (telephone: Bristol 27304). The Domestic Appliance Service Depot remains at 155 Whiteladies Road, Bristol, 8.

B.W.R.A. Open Day Research on Welding Processes and Welded Products



Sir Alexander Fleck (facing camera) with the Director Dr. R. Weck (left) in the Fatigue Laboratory.

IT has been customary for some years past for the Open Day at the British Welding Research Association's Laboratories at Abington Hall, near Cambridge, to be held annually, but this year's was the last until 1961, and a large number of members took advantage of the opportunity afforded to see something of the work in progress. The Open Day was also the occasion of the Annual Luncheon, and in proposing the toast to "The Association," Sir Alexander Fleck, K.B.E., F.R.S., Chairman of Imperial Chemical Industries, stressed the importance of the role the B.W.R.A. has to play in influencing the development of welding technology in the United Kingdom.

"The variety of industries served by the Welding Research Association is probably as wide as that served by any of the other forty-eight such bodies, and I am sure," said Sir Alexander, "that this is both a strength and a weakness. It is a strength because the Association can cast its net wider afield for its financial resources, and also because the important advances that are made by the Association can find wider application; but at the same time it is a weakness because such diffuse contact with industry in general is not conducive to the development of any kind of corporate spirit. In other words, only the manufacturers of welding equipment can speak of the B.W.R.A. as 'our research association'; the users, who form the bulk of the membership, are not, on the other hand, an easily definable group, covering as they do about twenty industries, so with them the sense of belonging to the Association cannot be so highly developed. As a result of this, it is doubtful whether the Association receives as much support from some quarters as it should.

"I know of no reliable way of estimating the number of firms who ought to support the work of the Association, and how much they should give," continued Sir Alexander, "but certainly the engineering, shipbuilding, electrical goods, metal manufacturing, vehicle and chemical industries should be well represented, as should the nationalised industries. Taken together, these

categories are estimated to spend over £170 m. annually on research and development, i.e. private and government sponsored, and if only one-thousandth part of this sum were given to the Association, it would get what it needs from industry to discharge its responsibilities under its new five-year plan. This is surely not an extravagant sum for these industries to set aside, bearing in mind the tangible benefits which accrue from systematic welding research.

"When one considers that in the United States the Welding Research Council spends about a million dollars a year on basic research alone, one realises the danger that Great Britain runs of falling far behind in welding technology. We may be quite sure, too, that the Russians are spending large sums in this field, and are also very active in teaching their engineers welding technology. Against this background it is clear that the B.W.R.A. has a particularly important role to play in influencing the development of welding technology in the United Kingdom, and doing so with resources which it recognises as inadequate to meet the country's needs."

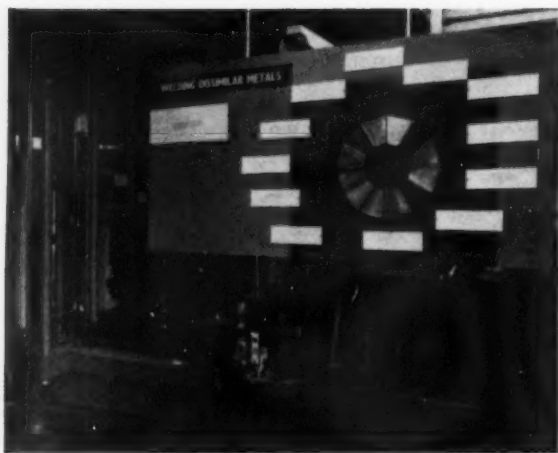
EXHIBITION OF WORK IN PROGRESS

For the benefit of visitors, interesting displays of exhibits designed to present a cross-section of the work in progress were staged in the appropriate laboratories, where members were afforded the opportunity to discuss with members of the staff the problems under investigation. In the following pages, brief reference is made to some of the items which may be of interest to readers.

Welding Process Laboratory

This laboratory is concerned with investigations of welding processes, and houses a comprehensive range of fusion welding equipment, provided with adequate instrumentation and capable of automatic operation wherever possible. Additionally, a large number of members' problems relating to the selection and use of welding processes is handled each year.

Considerable interest is at present being shown in



Part of the Metallurgical Laboratory with an exhibit illustrating the joining of dissimilar metals.

the bare wire CO_2 process for welding steel, and such aspects as spatter, recovery of alloy additions, and weld properties are being investigated in relation to welding conditions. Low voltages and high currents produce optimum spatter results, and high currents result in better recovery of elements in the filler wire: they also tend to increase the carbon content by pick-up from the gas shield. The mechanical properties of CO_2 shielded welds made using a filler wire deoxidised with aluminium and titanium, compare favourably with welds made in pure argon and argon-oxygen mixtures.

The way in which metal is transferred from the wire to the weld pool has been studied by the oscillographic examination of arc voltage. Current and wire diameter mainly control the size of particle transferred, although arc voltage is not without influence because with a short arc the size of droplet which can be formed is limited. Spatter is much reduced by operating with a short arc, but a power source of flat characteristic is necessary to be able to run an arc at the low voltages required. The short circuit current must be controlled to avoid the explosive fusing of droplets from the end of the wire, which can cause splashing and spatter.

In an investigation into the welding of sheet thinner than 14 s.w.g. by the CO_2 process, the problems of unfavourable metal transfer, spatter and irregular bead appearance have been solved, and welds made in a variety of materials, including low alloy and stainless steels, and Nimonic alloys.

Research is in progress to determine the factors controlling the transfer of droplets from an electrode across an arc, with a view to increasing the range of gas shielded welding processes. Using a specially constructed precision welding head, the relationships between current and burn-off rate; current, voltage and particle transfer frequency; and voltage, current and feed rate are being determined for iron, magnesium, copper, nickel, titanium, zirconium and alloys of these elements.

The change from spray transfer to globular transfer in the gas shielded welding of aluminium depends on the diameter of the electrode wire being used. The wire feed-rate/welding-current relationship has been determined for aluminium wire from $\frac{1}{8}$ in. to $\frac{1}{4}$ in. diameter.

On the assumption that welding cannot generally be done with globular transfer, this information has been defined for aluminium the lower working limit of the inert gas metal arc process. With wire of $\frac{3}{32}$ in. diameter and less, it appears that sufficiently low welding currents can be used to weld sheet material which it was previously considered impossible to weld. A hand torch developed by the Association to feed fine wires is being used to explore the welding of thin aluminium and steel sheet.

General Metallurgical Laboratory

As part of a long term investigation into the weldability of alloy steels, steel specimens are charged with hydrogen at 950°C . in a small furnace and quenched in a stream of argon gas. The quenched specimens are tested to fracture, and the drop in properties due to hydrogen has been found to be more severe in sheets which have a greater tendency to heat-affected zone cracking. The effects of hydrogen content and severity of cooling on heat-affected zone cracking are thought to be linked by a hyperbolic relationship, but this cannot be determined until a more complete range of hydrogen contents has been achieved in the weld metal. Bare wire welding in an atmosphere of argon with controlled humidity is being used to obtain weld metal with a hydrogen content intermediate between the values given by rutile covered and low-hydrogen electrodes. A new wedge test for heat-affected zone cracking provides a range of cooling severities in one specimen and, although it is not intended to replace the C.T.S. (controlled thermal severity) test, it is simpler and will speed up research.

In studying the mechanism of cracking in fully austenitic weld metal, a knowledge of the temperature at which cracks form would be very useful. The Association has developed apparatus for obtaining this information by simultaneous measurement of the temperature and electrical resistance of a cooling weld nugget. The ductility of austenitic steels at high temperatures is being examined as part of an investigation into the problem of parent metal cracking of weldments. Ductility, temperature curves, plotted from torsion tests carried out on heating to and cooling from a temperature close to the melting point, have shapes which appear to bear a relationship to the known tendencies of the materials to cracking. With the increasing use of nuclear power stations to meet base load requirements, present stations having austenitic steam piping may be shut down overnight, with resultant cyclic application of stress to parts of the steam plant. Conventional creep testing equipment has been adapted so that a specimen can be subjected to a variable strain cycle at high temperature to simulate operating conditions.

There are advantages to be gained by welding domestic hot water cylinders rather than brazing them, but argon arc welding of thin gauge phosphorus-deoxidised copper without filler wire results in porosity. The Association has shown that success may be achieved by substituting zinc-deoxidised ("cap") copper for the phosphorus-deoxidised material. At present the former is more expensive, but its use on a large scale would probably eliminate this disadvantage. Both argon and nitrogen have been used as the shielding gas in the consumable electrode welding of copper, the former giving smoother metal transfer and the latter greater penetration. By mixing them the advantages of each can be combined, a suitable compromise containing 20-30% nitrogen.

Still in the copper field, the problems of cracking and

embrittlement after multi-pass welding single phase aluminium-bronze are being investigated by studying the high temperature strength and ductility of various weld metal compositions following a thermal cycle designed to simulate actual welding conditions.

The Association has recently completed its work on titanium, in the course of which particular attention was given to such factors as the mechanism of gas shielding when welding in the "open air", and the effect of contamination by oxygen and nitrogen, both when present in depth in the weld metal, and as a surface effect. Exhibits relating to the welding of reactive metals—such as titanium and zirconium—included reference to the effect of gases on reactive metals; the mechanism of the development of porosity in butt-welded titanium; gas shielding equipment for welding reactive metals in the "open air" and equipment for welding in a chamber filled with shielding gas.

Weld metal deposited in certain ways has a high ratio of yield to ultimate strength, and is also stronger than cast or wrought metal of the same composition. The reason is of intrinsic interest in welding research, because if the mechanism were understood, the effect could possibly be controlled to advantage. Weld metal deposits made by various welding processes are being examined for their mechanical properties and by electron microscopy.

Resistance Welding Laboratory

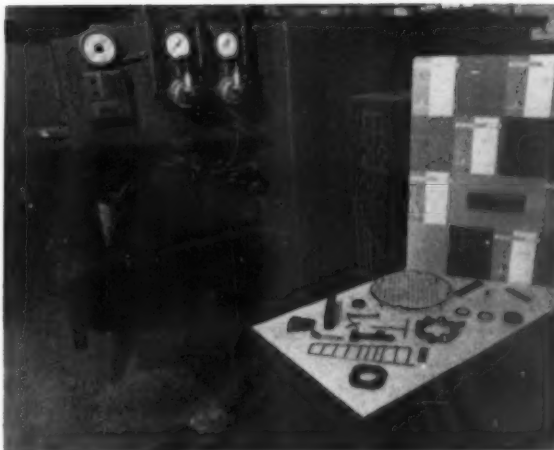
When steels with carbon contents exceeding about 0.15% are spot welded, a hard brittle structure is formed as a result of the high quench rate inherent in the process. Investigations in progress are aimed at a reduction in the large amount of experimental work normally necessary to determine the best post-weld heat treatment to apply in the spot welding machine to improve the ductility at the spot weld. The measurement of non-sinusoidal currents; the study of temperature cycles in spot welds; the determination of suitable tests for weld ductility; and the assessment of metallurgical structures produced by post-weld heat treatment have all required consideration. During the past year, the main effort has been directed towards the last of these, and the effects of such heat treatments on spot weld ductilities have been correlated with the structures produced.

The resistance stud casting process was again displayed in the hope that further suggestions for its application in industry would be forthcoming. The process is based on the discovery that metal may be expelled from a spot weld nugget into a mould on the electrode, where it is cast to form a stud on the weldment.

An interesting exhibit in this laboratory concerned an investigation carried out under contract. The problem arose from the necessity to measure temperatures within nuclear fuel elements. It involved the making of a leak-proof junction between the magnesium alloy lid of a nuclear fuel can and the stainless steel sheath of a thermocouple cable. The various stages in making and testing the joint were shown.

Fatigue Laboratory

In a study of the influence of fabrication technique on the fatigue strength of welded thin-gauge heat resisting alloy components, such as are used in jet engines, tests are being made on manual and automatic argon arc



Part of the Resistance Welding Laboratory showing some of the jobs that have been carried through to assist members during the year.

welds in Nimonic 75, 80A and 90. For a given sheet thickness, the variables are testing temperature, stress cycle, post-weld heat treatment, welding process and surface treatment. Other work on these alloys includes a comparison at room temperature of two series of spot welds in Nimonic 75 sheet, with and without "radiographic" defects such as small cracks and cavities. Because of the dominant stress concentration effect of the edge of a spot weld, both the modes of failure and the strengths were similar for the two series. Argon arc welds in commercially pure titanium have been tested in order to observe the effect, if any, of surface contamination (oxidation) on tensile fatigue properties. Some fatigue data for argon arc welded titanium alloy (5% Al, 2½% Sn) have also been obtained.

In the light alloy field, the influence of reinforcement shape on the fatigue strength of butt-welded NP5 6 aluminium alloy plate is being investigated. The automatic self-adjusting arc process is being used in this work in order that the reinforcement shape can be varied in a controlled manner. Where no particular precautions are taken to control this shape, tests on NP5 6 and H30 plate, welded by the argon arc, self-adjusting arc and metal arc processes have shown that the welding process is not, in itself, a critical factor in determining the fatigue strength. Preliminary fatigue tests on plain specimens of H15 in the clad and unclad form have shown that the cladding lowers the fatigue strength appreciably, and it is expected that similar differences will exist after welding.

In order to help in assessing the seriousness of defects in welded joints revealed by non-destructive testing, fatigue tests are being made on such joints. What look like major defects often have little effect in tensile testing, and it is felt that fatigue testing will be more severe. As a preliminary to this work, techniques have been established for the controlled production of such defects as slag inclusions, porosity and lack of penetration.

The failures which sometimes occur soon after the return to service of worn shafts which have been reclaimed by welding are usually associated with a condition of dynamic stressing. The work planned in a study of this



A view of the Fatigue Laboratory showing 100 ton fatigue machine, high pressure fatigue machine, and tests on stiffened beams.

problem makes provision for a study of pre-and post-welding heat treatment. Tests to date suggest that the presence of defects might interfere with the comparison of heat treatments, as failure has usually originated in isolated defects at or near the surface: good results are obtained when such defects are absent.

Fatigue tests on plate specimens representing part of a water tube boiler, and containing an array of holes in which diameter, pitch, longitudinal spacing and rake can be varied, have shown that there is good correlation between fatigue behaviour and stress distribution as indicated by photoelastic analysis. The results are expected to form a basis for reviewing the present-day design methods employing mean ligament stresses. Other fatigue tests in progress on parts of structures rather than normal test pieces include the pulsating pressure tests on thick-walled pipe specimens containing circumferential butt welds, and the study of the damaging effect of attaching a web stiffener to the tension flange of a beam.

In cases where the fatigue strength of a welded joint is determined by the presence of a localised notch, as with discontinuous longitudinal welds, large increases in fatigue strength may be obtained by inducing residual compression at the notch, either by spot heating or local mechanical compression. Examples of the application of this principle to service problems were shown, and it was demonstrated that the two methods also increase the fatigue strength of suitable details in aluminium alloys.

Pressure Vessel and Pipe Line Laboratory

The immense size of nuclear reactor pressure vessels, and the impossibility of routine inspection, necessitate a more critical estimate of strength than is customary for pressure vessels of conventional size. Furthermore, certain features, such as fuel charge nozzles and cooling gas duct entries, are not capable of satisfactory traditional design. Some calculations have been made of the stresses due to pressure at duct entries, and experimental

stress analysis of models of fuel charging nozzles have been made.

Pulsating pressure tests of nozzle details have been performed on 20 in. bore pressure vessels, and the results have confirmed that the traditional method of strengthening a branch connection by means of a welded external collar does little to improve the resistance to fatigue failure, which is, however, extremely unlikely whether the connection is reinforced or not. Stress measurements round unreinforced nozzles of a very thick drum have confirmed the prediction that the stress concentration factor for flush-fitting, unreinforced nozzles would be smaller in a drum of large thickness/diameter ratio. Whilst some increase in design stress would be tolerable, without any reduction in the stress concentrations existing at unreinforced nozzles, it would be desirable to achieve the maximum increase by a small improvement in nozzle detail design. The results of stress measurements on the protruding nozzle show that this is one design modification which diminishes stress concentrations.

As a rational alternative to the conventional pad reinforcement of manholes, the use of tubular rim reinforcements has been investigated. Such designs show a great economy in material and welding, and as stress calculation methods have been found to agree with measurements on quarter-scale models, design dimensions have been calculated for a range of manhole sizes, for various values of stress concentration factor.

The value of the frozen stress technique in the determination of stresses at details in pressure vessels is being explored initially by obtaining photo-elastic analyses of components similar to the steel constructions tested earlier. This will permit a comparison of photo-elastic and strain gauge measurements. It is expected that the method will prove more rapid and no less accurate than strain gauge measurements, and will permit results to be obtained for a wider range of variables.

When furnace treatment is not possible, circumferential welds in pressure vessels or pipes are sometimes stress relieved by applying heat to a narrow band around the vessel. The temperature gradient along the vessel induces thermal stresses, which may cause yielding of the hot metal near the weld. On cooling, a secondary residual stress system may appear, which has no relation to the original residual stresses due to welding. A logical choice for the heating conditions would be one which does not induce severe thermal stresses, and in order to make this choice possible an approximate relationship between the thermal stress and the width of the heating band has been determined.

Brittle Fracture Laboratory

Brittle fracture is known to have been occurring for over a hundred years, and although no class of structure is exempt, catastrophic failures may occur in welded structures because they provide a continuous path for the crack. Research work carried out by the Association in the last few years has established that, for brittle fracture to occur: (a) the temperature must be below that at which a given type of steel fractures in this way (the transition temperature); (b) there must be some

notch effect; and (c) there must be residual stress in the structure: unless all these conditions prevail, brittle fracture cannot occur. The main safeguard, in addition to a design which eliminates notch effects, is to relieve the residual stresses after welding should the structure be too large, the only sure way of avoiding the risk of brittle fracture is to choose a steel with a transition temperature below that likely to be encountered in service.

For some years B.W.R.A. has been testing butt welded steel plates 1 in. thick and 36 in. wide, but reactor vessel design calls for the use of 3 in. thick plate. To meet these requirements, there is now available a testing machine designed by B.W.R.A. and presented to the Association by Babcock & Wilcox, Ltd., which is capable of applying a load of over 2,000 tons to fracture 36 in. wide by 3 in. thick welded plates. Because of the larger grain size, thick plates will frequently break in a brittle manner at higher temperatures than thin ones of the same composition.

A comparison between low-hydrogen and rutile-coated electrodes for welding notched wide plate specimens shows that the latter, with a greater tendency for cracking across the weld at the notched zone immediately after welding, give lower brittle strengths at given temperatures.

Crack extension force (also called strain energy release rate) is the quantity which determines the possibility of cleavage crack propagation. It has recently been shown by Irwin to be uniquely related to the stress distribution around the tip of the crack, and so may now be measured by photo elasticity.

By making use of all the knowledge obtained from B.W.R.A. tests and that at present possessed by the steelmaking industry, it is now possible to produce welded structures in 3 in. thick plate possessing a high degree of resistance to brittle fracture. This is a notable contribution to the development of nuclear power generation.

The Corrosion of Iron and Steel

Twenty Years' Research Reported by Corrosion Committee

THE Corrosion Committee, formed in 1928 with Dr. W. H. Hatfield, F.R.S., as its first Chairman, has now been experimenting for more than thirty years, and the Sixth Report of the Committee, published by The Iron and Steel Institute, contains an account of its researches during the last twenty years of this period. The Report covers the corrosion of iron and steel in air, in soil, and in water, details the results of numerous experiments, and recommends methods of protecting ferrous metals against atmospheric corrosion, soil corrosion, marine corrosion, and corrosion by industrial waters.

A particularly interesting aspect of the experiments consisted in finding out the corrosion rates of different kinds of bare steels in different geographical locations. For example, tests have been made in the polluted air of industrial Sheffield, in the sea air of Calshot, and in the relatively pure country air of Llanwrytyd Wells. The rates of corrosion of irons and steels in the soil have also been determined by burying specimens at various places for periods of from five to fifteen years, and immersion tests in the sea have been made by suspending specimens from rafts.

Atmospheric Corrosion

The atmospheric field tests have given a clear insight into the effects of differences in the compositions of structural irons and steels on their corrosion rates in the bare condition. In particular, the addition of fractional percentages of alloying elements such as chromium, copper, and nickel to mild steel increases its resistance to atmospheric corrosion appreciably; some low-alloy steels containing chromium and copper are at least three times as resistant to the atmosphere as unalloyed mild steel. The differences between the various steels are, however, much less marked, even non-existent, when they are immersed in water or buried in the soil.

Parallel tests have been made with steels protected by paint, or metal coatings, or both. In the case of paint,

a very significant finding is that the life of the protective coating does not generally depend on the corrosion rate of the steel, but primarily on the surface preparation given to the metal before it is painted. Many different methods of surface preparation have been compared. For atmospheric work the best was found to be to descale the steel by pickling or blasting, and to apply the priming coat of paint immediately afterwards.

The problem of selecting the best priming paints is complicated, and involves choosing the best combinations of pigments and paint media with due regard to the material to be painted and the method of surface preparation to be adopted. Field tests on priming paints for steel have been in progress for some time, in collaboration with the paint industry. In one set of experiments, tests are being made on a series of 100 priming paints in which each of a range of pigments is bound with each of several different kinds of medium. In other sets 91 priming paints containing metallic pigments, and 55 priming paints based on bitumen and tar, are being tested.

The protection given to iron and steel by paint is materially improved by the additional use of coatings of non-ferrous metals, less susceptible to corrosion, beneath the paint. This method of protecting steel is being used increasingly on bridges and other structural steelwork. In order to be able to give guidance on the technical problems involved in the use of these composite protective schemes, the Committee has undertaken an investigation into the suitability of different paints for use over metal coatings. These tests are being made on steels which are first coated with aluminium or zinc by spraying, and then painted.

"High" Temperature Corrosion

The Report contains an account of investigations undertaken during the Second World War into the corrosion and protection of steel at temperatures up to 350° C. These arose in connection with the protection of steel sheets used for anti-glare structures, which were

exposed under sheltered conditions over pig-beds and coke-ovens to highly corrosive atmospheres polluted with steam and sulphur gases. In these circumstances, no significant improvement resulted from using low-alloy, copper-steels. Even the best painting schemes failed to provide adequate protection at temperatures much above 250° C. Vitreous enamel would probably have served at these higher temperatures, but under the prevailing wartime conditions the only practicable solution was to use the thickest sheets available and allow them to rust to destruction.

The corrosion of buried pipes is a major source of trouble in industry, and in an effort to solve some of the problems of soil corrosion the Committee has carried out numerous field tests with different irons and steels, and has tested the efficiency of various types of protective coating, notably hot-dipped tars and bitumens.

Marine Corrosion

Marine corrosion is a particularly important aspect of the Committee's research. Immersion tests in sea water have been made on both bare and coated steel, and the results for bare specimens have shown that small variations in the composition and structure of mild steel, such as result from different manufacturing processes, have no significant effect on its corrosion resistance. The addition of 3% of chromium roughly halves the corrosion rate over a period of five years, but in general low-alloy additions do not materially affect the corrosion rate, as they do in the case of atmospheric exposure.

Extensive research has been devoted to the development of paints for underwater use. As a result, several reliable anti-corrosive compositions for ships' bottoms have been formulated, some of which have given satisfactory service on H.M. ships and the "Queens" for many years.

In parallel with the raft experiments needed for this work, service trials have been made on the bottoms of large ships. These have shown, for example, that there is no foundation for the belief that launching a vessel without painting the bottom will help to improve the performance of paint applied subsequently.

Fundamental Studies

In conjunction with the practical work that has been described, the Committee has paid considerable attention to fundamental studies of the theory and mechanism of corrosion, and to the development of laboratory corrosion test procedures. In the former field, it has had the valuable support of Dr. U. R. Evans, F.R.S., and the Cambridge school of investigators; a summary of their work concerned with the corrosion of ferrous metals is given in the Report.

As regards corrosion testing procedures, the Committee's investigations have revealed that it is improbable that any satisfactory all-embracing laboratory corrosion test can be found. Laboratory tests are, however, of considerable usefulness for specific purposes and the Committee has assisted the British Standards Institution in checking the validity of two laboratory corrosion tests. These are the A.R.E. Salt Droplet Spray Test and the C.R.L. Sulphur Dioxide Beaker Test, both of which have been standardised by the Institution. Other work on laboratory test procedure has been concerned with the reliability of various types of non-destructive testing instruments for determining the thickness of metal coatings on steel.

The Report is not, of course, final, because the Committee's work is continuing and, apart from this, the normal course of industrial progress will continue to produce new and better methods of protection. The Report does, however, represent an up-to-date exposition of knowledge of many aspects of corrosion of ferrous metals. As such it is an invaluable contribution to industry and should be read by anyone interested in problems of corrosion.

Process Control in Oxygen Steelmaking

A SPECIAL automatic control system will monitor an unusual steelmaking process when the Acme Steel Company's furnaceless plant begins production soon in Chicago. The system, developed by the American associates of Honeywell Controls, Ltd., will regulate and total the flow of oxygen to the converters. It will also monitor and control the temperatures of the converter exhaust gases so as to safeguard the precipitators through which these pass for removal of impurities. The £10 million plant—first steel-producing facility of its type in the U.S.—will use hot-blast cupolas to melt the iron, which will be made into steel by oxygen converters. The only other plant of this type is in Germany, where the process was pioneered in 1957.

Each "heat" of steel will require different amounts of oxygen, depending upon the composition of the charge—the molten iron—and the grade of steel to be produced. Totaliser controls, which will keep a precise check on the flow of oxygen to see that the right amount is delivered to the converters, will be applied first. The custom-designed control systems—one for each of the plant's two oxygen converters—are housed in two cubicle-like steel rooms, and among major components of the systems are electronic devices that constantly measure the flow of oxygen to the converters, and of water to cooling towers where the exhaust gas temperatures are lowered. This process information will be transmitted to separate recorder-controllers, which will compare the measurements against desired values. Any differences between them will result in the pneumatic positioning of oxygen and water line valves so that correct flows are maintained.

Proponents of the furnaceless steelmaking process claim that a superior grade of steel can be made faster and at less cost than by conventional methods. Acme Steel has been a steel fabricator for 79 years, but the start-up of the furnace-less plant will mark its entry into the producing field. Projected initial capacity of the new plant will be 450,000 ingot tons annually, which will provide 70% of Acme's requirements of billets and slabs for its own fabricating work.

Hilger & Watts in the North

HILGER & WATTS, LTD., makers of instruments for analysis, surveying, photogrammetry, metrology, and industrial inspection, have appointed a new representative, whose area will be Yorkshire, Lancashire, Cheshire and North Lincolnshire. He is Mr. D. G. HEYWOOD, 21, Kirkstall Road, Sheffield 11 (tel: Sheffield 64226). Mr. Heywood's particular province will be the firm's analytical instruments: spectrographs, polychromators, spectrophotometers, polarimeters, refractometers, absorptiometers, colorimeters, etc.

Fretting Corrosion of Metals

By R. T. Allsop, B.Sc., Ph.D.

(G.K.N. Group Research Laboratory)

Damage resulting from fretting corrosion is observed in a variety of engineering components including bearings, switchgear and bolted or riveted joints. The present review examines first the nature and occurrence of fretting corrosion and then discusses the various theories which have been proposed to explain the phenomenon. In a further section the effect of varying such factors as humidity, duration, load, etc., on the amount of damage occurring during fretting is considered. Finally methods of eliminating or reducing fretting damage are suggested.

(Continued from page 43 of the August issue)

Influence of Various Factors on Fretting Corrosion

Duration

The results of Feng and Uhlig¹¹ and Wright⁸ confirm quantitatively the common experience of engineers that fretting damage increases with the duration of the test when it is conducted in air. Although the above authors used different methods of measuring fretting damage, and somewhat different experimental conditions, it is of interest to compare the general form of the damage/time (number of oscillations) relationship obtained in each case. Fig. 3 shows the type of curve obtained by Feng and Uhlig and Fig. 4 that produced by Wright. It can be seen that the latter is approximately linear and passes through the origin, whereas the former, although eventually becoming linear, shows that damage commences at a rapid rate and then decreases to a constant rate. Feng and Uhlig have referred to the initial stage of their experiments as a "run in" period.

Although, from a practical point of view, the relationship between damage and duration over relatively long periods is probably the more important, there is considerable theoretical interest in the form of the damage/duration curve in the initial stage of the fretting process.

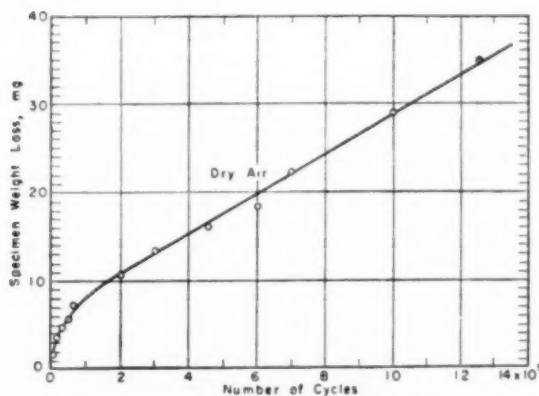


Fig. 3—Effect of duration of test on fretting of mild steel in dry air (pressure, 5,300 lb./sq. in.; frequency, 540 c./min.; slip, 0.0036 in.; average temperature, 33° C.) (after Feng and Uhlig¹¹).

As mentioned previously, Feng and Rightmire have recently examined in detail the initial stages of fretting between steel surfaces in a variety of atmospheres. The results obtained in dry air are shown in Fig. 5, from which it can be seen that, contrary to the monotonic curve proposed earlier by Feng and Uhlig, there is, in the early stages, a plateau during which little additional damage takes place. The explanation of this discontinuity was considered in the earlier discussion of the theories of fretting corrosion.

Composition of Atmosphere

Several investigators have conducted fretting experiments in atmospheres other than air. In a dry nitrogen atmosphere, Feng and Uhlig¹¹ have observed (Fig. 6) that the damage which occurs is considerably less than that occurring in air. Using an atmosphere of dry CO₂, Feng and Rightmire⁷ found (Fig. 7) that, although the plateau period in the early stages of fretting was much extended, the total damage occurring over long periods

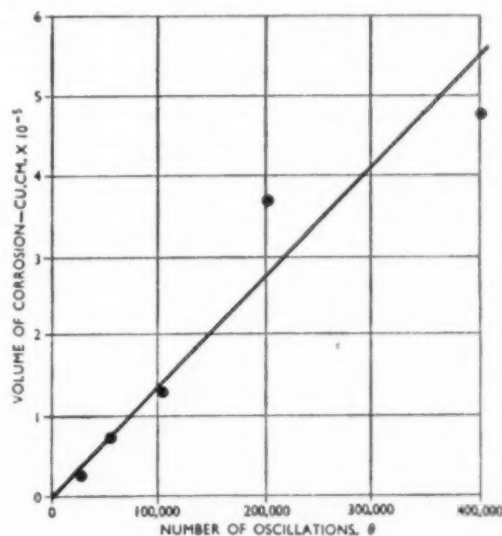


Fig. 4—Variation of volume of fretting corrosion (estimated chemically) with number of oscillations (after Wright⁸).

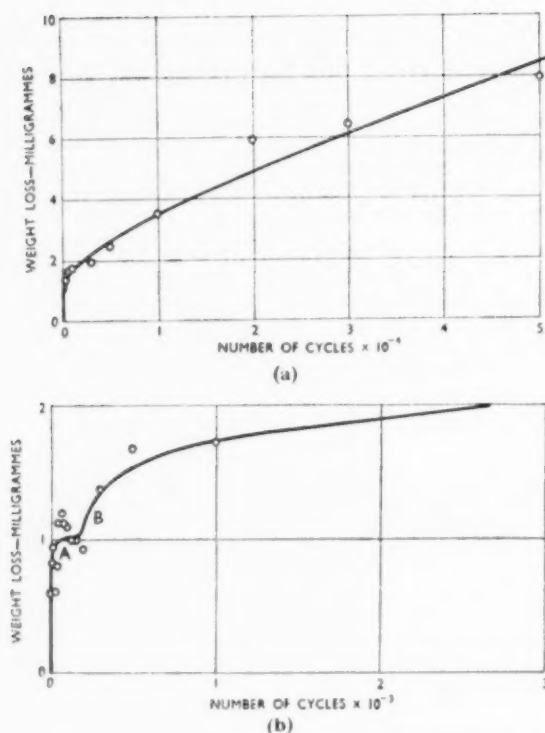


Fig. 5—(a) Curve of fretting weight loss against number of cycles in dry air; (b) part of the same curve near the origin, enlarged to show its shape more clearly (after Feng and Rightmire⁷).

approximated to that occurring in air. Feng has suggested that these results illustrate clearly that the concept of corrosion is misleading in connection with damage produced by fretting. He argues that the concept of fretting as a corrosion process would necessitate damage being virtually eliminated in a non-reactive atmosphere. If, therefore, it is accepted that CO_2 is an inactive atmosphere, then the damage occurring should be much less than that in air, rather than of the same order.

In order to explain the differing effects of various inert atmospheres, Feng and Rightmire suggest that whether, in fact, the rate of damage ever accelerates from the plateau region depends on the ability of the atmosphere to react chemically with the metal to form a surface layer, or to form an adsorbed layer which can interfere

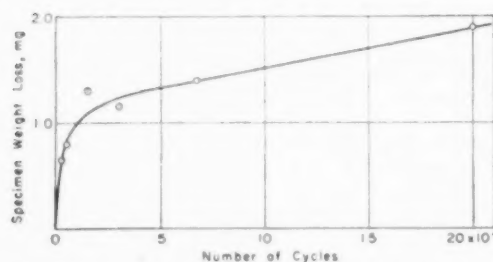


Fig. 6—Fretting of mild steel in nitrogen (after Feng and Uhlig¹¹).

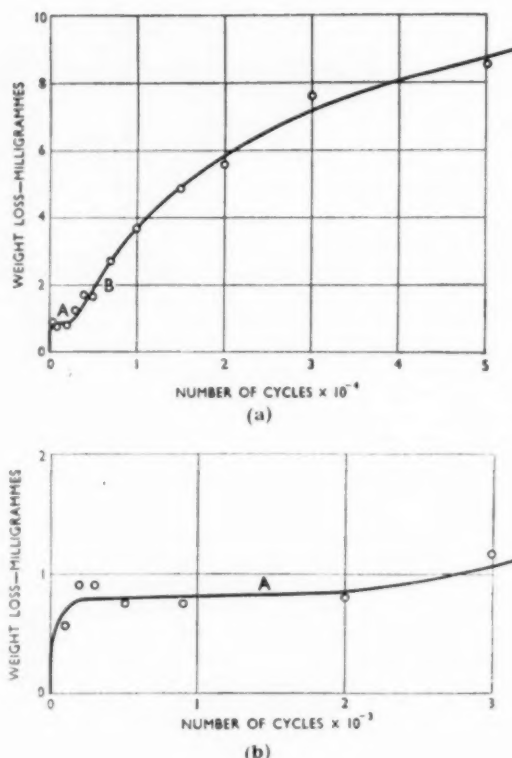


Fig. 7—(a) Curve of fretting weight loss against number of cycles in carbon dioxide; (b) part of the same curve near the origin, enlarged to show its shape more clearly (after Feng and Rightmire⁷).

with metal transfer. In the case of CO_2 the amount of gas adsorbed is sufficient to permit the eventual generation of the free particles which allow the fretting process to continue by abrasion. Nitrogen and helium, on the other hand, form only slight adsorbed films; this means that experiments in such atmospheres will probably never develop beyond the "plateau" stage.

Humidity

The considerable influence exerted by the humidity of the air in which a fretting corrosion experiment is carried out has been noted by several investigators. Wright⁸ has found (Fig. 8) that when the humidity is increased from zero the damage for steel on steel initially decreases, but that above 40–60% humidity the damage begins to increase again. For chromium against steel, the fretting damage decreases progressively as the humidity increases from zero to 100%. Feng and Uhlig¹¹ have reported that for steel against steel the amount of fretting damage decreases with increasing relative humidity (Fig. 9). Results at 100% humidity were not obtained in this investigation, because of the rusting which occurred as a result of condensation of water on the specimens. As an explanation of the effect of variations in humidity on fretting damage, Wright has suggested that the water adsorbed or deposited by capillary condensation on to the metal and oxide surfaces in humid atmospheres acts as a

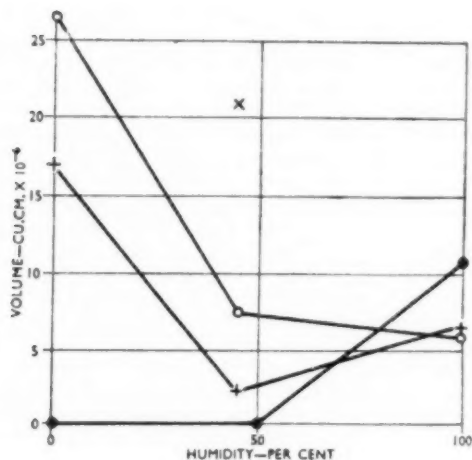


Fig. 8—Variation of fretting corrosion with humidity (after Wright⁹).

lubricant which promotes the removal of debris from contact areas. Such a mechanism appears to be in agreement with the observation that the amount of fretting damage increases sharply when the temperature is reduced to the freezing point of water.

The data available on the influence of humidity on fretting damage in atmospheres other than that of air is limited; only Feng and Uhlig appear to have investigated this point. They found that a wide variation in humidity had little or no effect on the amount of damage occurring in a nitrogen atmosphere.

Normal Load

The effect of varying the normal load applied between fretting surfaces on the amount of damage occurring is complicated by the fact that as the normal load is increased so the magnitude of the relative slip decreases. If no attempt is made to maintain the slip at the original value, the variation of fretting damage with load is given by the results of Uhlig, Tierney and McClellan,²³ from

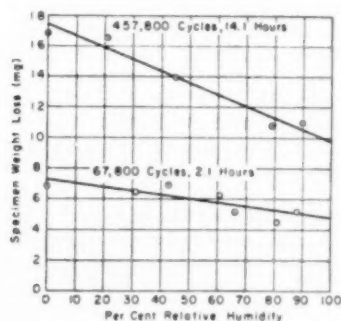


Fig. 9—Effect of humidity on fretting of mild steel for two test periods in air (pressure, 5,300 lb./sq. in.; frequency 540 c./min.; slip 0.0036 in.; average specimen temperature, 33° C.; room temperature, 27° C.) (after Feng and Uhlig¹¹).

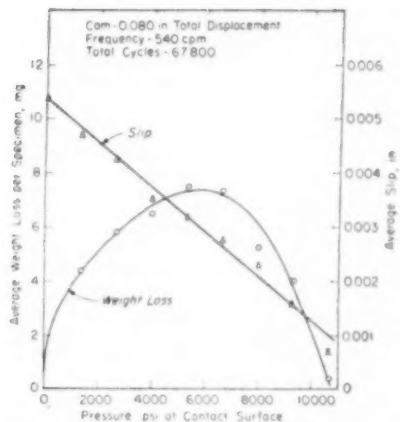


Fig. 10—Effect of pressure (load) on weight loss and slip (after Uhlig, Tierney and McClellan²³).

which it can be seen (Fig. 10) that as the load is increased from zero so the damage at first increases; with further increase in load, the progressive decrease in the amount of slip occurring begins to reduce the amount of damage until finally, when relative movement is completely eliminated, damage ceases.

If, as the load is increased, the slip is maintained constant by adjustment, the variation of fretting damage with load is given by the results of Wright⁸ who showed (Fig. 11) that fretting damage increases in almost linear manner as the normal load is increased. Similar results have been obtained by Feng and Uhlig.¹¹

Frequency

Frequency of oscillation as a variable in fretting corrosion has not been studied extensively, although several authors have commented, without detailing experimental results, that variations in frequency over quite a wide range have no effect on the amount of damage occurring. In contrast, Feng and Uhlig have carried out fretting experiments at various frequencies and degrees of slip;

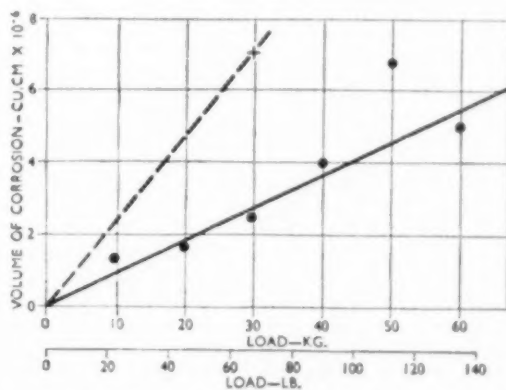


Fig. 11—Variation of the volume of fretting corrosion (estimated chemically) with load. Dotted curve—humidity 100%; full curve—humidity 50% (after Wright⁹).

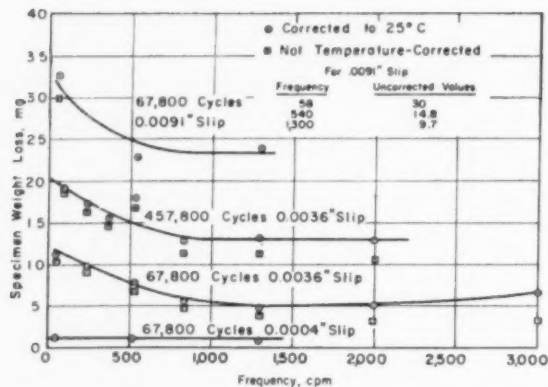


Fig. 12—Effect of frequency on fretting of mild steel in dry air (pressure, 5,300 lb./sq. in.) (after Feng and Uhlig¹¹).

they showed that for relatively large amounts of slip frequency does influence the amount of damage produced by fretting. The nature of the effect is shown in Fig. 12. Variations in temperature produced by changes in frequency were a complicating factor, and Feng and Uhlig made an attempt to correct their damage values to a common temperature, 25°C.

Amplitude of Relative Motion

The various reviews and original investigations of the subject of fretting corrosion have repeatedly reaffirmed the conclusion of Tomlinson *et al*¹ that relative slip is essential if fretting damage is to occur. A study of the effect of variations in amplitude on the magnitude of fretting damage has been carried out by Feng and Uhlig¹¹; their results (Fig. 13) showed that over the range of slip examined the damage resulting was directly proportional to the amplitude of slip.

Many of the investigators studying the phenomenon of fretting corrosion have emphasized the need for distinguishing between "real" and "apparent" slip. Apparent slip is, essentially, the relative movement which would occur if the contacting components were made from completely rigid materials. In practice, of course, some of the enforced movement is taken up as elastic strain in the contacting surfaces; the "real" slip is, therefore, the

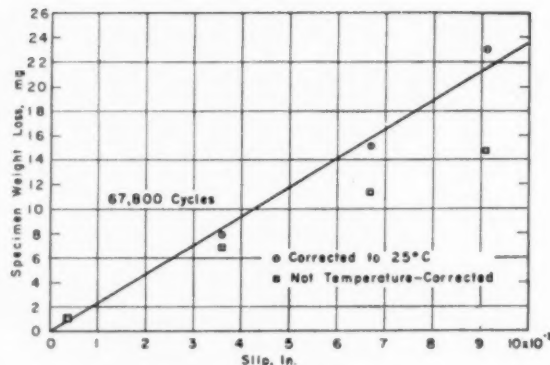


Fig. 13—Effect of relative slip on fretting of mild steel in dry air (pressure, 5,300 lb./sq. in.; frequency, 540 c./min.) (after Feng and Uhlig¹¹).

difference between the apparent slip and that absorbed as elastic strain in the material. The more theoretical aspects of the relationship between real slip and elastic movement have been considered by Johnson.²⁴

Temperature

Although it has been known for many years that seasonal variations in ambient temperature influence the amount of damage occurring during fretting, a detailed study of the effect has only recently been made (Feng and Uhlig¹¹). The results of this study (Fig. 14) show that, as the temperature falls below 0°C., damage increases for long periods of test, but remains relatively fixed for shorter periods. Increasing the temperature above 0°C. produces a relatively rapid decrease until 50°C. is reached, after which temperature has little effect on the amount of damage produced.

Nature of Debris Produced in Fretting

The composition of the debris produced during fretting varies considerably with the prevailing experimental conditions. Several investigators,^{8,25} have confirmed that in moist air the debris produced during the fretting of steel is the α -form of Fe_2O_3 . Under conditions of low humidity, Wright²⁶ observed that the debris still consists of α - Fe_2O_3 . Feng and Rightmire,⁷ on the other hand, whilst agreeing that the bulk of the debris produced under such conditions is Fe_2O_3 , contend that a measurable quantity of α -iron is also present. To resolve the apparent anomaly, Wright has suggested that in cases where relatively soft steels are used, the amount of α -iron in the debris will be considerably greater than where hard steels are employed.

Under conditions in which the supply of oxygen to the fretting surfaces was restricted, Wright⁸ found that both F_3O_4 and α - Fe_2O_3 were present, the latter only appearing however, after about 10^6 oscillations. When a fretting experiment was surrounded by a nitrogen atmosphere, Feng and Uhlig¹¹ observed that the debris produced was α -iron.

Prevention of Fretting Corrosion

The purpose of research into the mechanism of fretting corrosion and the factors which affect it is, primarily, to provide a foundation on which methods of preventing fretting damage can be based. Although many aspects

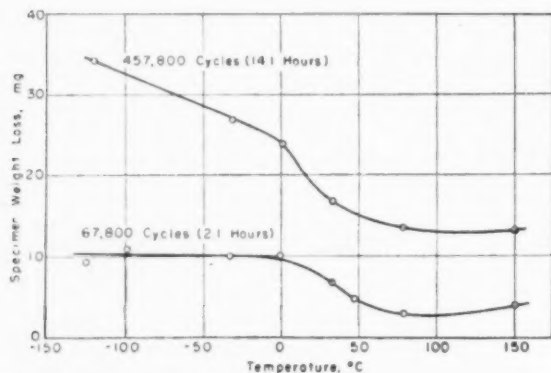


Fig. 14—Effect of temperature on fretting of mild steel for two test periods in dry air (pressure, 5,300 lb./sq. in.; frequency, 540 c./min.; slip, 0.0036 in.) (after Feng and Uhlig¹¹).

of the mechanism of fretting corrosion still require clarification, it is nevertheless possible to indicate various ways in which damage may be reduced or completely eliminated. It should be emphasised, however, that methods of alleviating fretting which may prove effective in one instance will not necessarily prevent damage in others; essentially, individual cases of fretting should be considered on their merits.

The techniques available for combating fretting are as follows:

Elimination of Vibratory Forces

The most obvious method of preventing fretting corrosion is to eliminate the vibratory forces generating the relative slip between the fretting surfaces. Whilst in some instances it is possible to design structures in which harmful vibration is reduced to a level below that necessary for damage to occur, prevention of fretting by this method is generally impracticable.

Elimination of Relative Slip

Instead of attempting to reduce the vibratory force causing relative slip, an alternative method of preventing fretting is to increase the frictional force opposing slip between the contacting surfaces. The frictional force is equal to the product of the normal load and the coefficient of friction; in order to increase the frictional resistance to slip, therefore, it is necessary to increase the normal load, the coefficient of friction, or both. If the normal load can be increased to the point at which slip is eliminated, then the method is fully effective in preventing fretting. On the other hand, as was seen earlier, if the load increase is insufficient to prevent relative movement completely, then the fretting damage may be increased. A practical example of preventing fretting by this means has been quoted by Gray and Jenny.²⁷

It was noted above that the relative movement producing fretting could also be eliminated by increasing the coefficient of friction. The method by which this can most easily be effected is probably that of plating the contacting surfaces with metal possessing the required frictional properties. Amongst the metal coatings reported as being successfully employed are silver, gold, tin and copper. Wright²⁸ noted that tin plating prevented damage to aluminium components, whilst Gray and Jenny²⁷ observed by laboratory experiment that copper was effective. Several investigators have noted that, unless the change in the coefficient of friction is sufficient to eliminate movement completely, then damage still occurs.

Exclusion of Atmosphere

In many cases the methods of completely eliminating fretting outlined above cannot, for one reason or another, be employed. When such a situation arises, there are several techniques by which the fretting can be markedly reduced, if not completely prevented. The preceding review of the experimental data has indicated at several points the considerable influence of the surrounding atmosphere upon the amount of damage occurring during fretting. It has been shown that when fretting occurs in the absence of oxygen, the damage is much less than that occurring when it is present. One possible technique of reducing fretting damage, therefore, is to surround the fretting components by a vacuum or an inert atmosphere. The exclusion of air by the introduction of vacuum conditions is scarcely a practical

undertaking, nor is the surrounding of the contacting surfaces by an inert gas such as helium. It would appear that the only method of reducing fretting by atmosphere control, is that investigated by Wright.⁸ The procedure adopted involves the introduction between the fretting surfaces of a liquid in which oxygen has a low solubility. Such a film mechanically excludes oxygen from the fretting area. Wright has demonstrated the efficacy of this technique by comparing the amount of damage occurring when B.P. paraffin and dried normal hexane are introduced between the surfaces. The introduction of paraffin reduces the damage by a factor of some 200 over that occurring in air, whilst the hexane reduces the damage to a much smaller extent. The difference in effect of these two lubricants is considered to be due to the greater solubility of oxygen in the hexane.

Lubrication

A second method of reducing fretting damage which may be employed where slip cannot be eliminated is that of reducing the coefficient of friction by means of lubrication. The theories of friction and wear, whether that of Bowden and Tabor¹⁶ or that of Feng¹⁷ indicate that when there is friction between two surfaces, there will also be damage to the surface in the form of either metal transfer or wear particles. It follows, therefore, that any method of reducing friction will also reduce surface damage.

The experiments of Campbell have led him to the conclusion that the principal reason why various hydrocarbon fluids reduce fretting damage is because they are able to effect separation of the fretting surfaces. Wright, on the other hand, inclines to the view that such fluids would be unable to effect separation under the severe conditions operating, and that their principal effect is one of excluding oxygen from the fretting zone. The effectiveness of normal extreme pressure lubricants to alleviate fretting is also considered doubtful by Wright, since it appears unlikely that the necessary temperatures required for the reaction of the lubricants with the surfaces will be attained.

Several investigators,^{13,8} have emphasised the usefulness of phosphating steel surfaces subject to fretting. Whilst such coatings are of little assistance in the dry condition, their ability to maintain a film of oil at the rubbing surfaces results in a considerable decrease in fretting damage. Schulman and Waterhouse²⁹ have observed that phosphate-coated steel specimens, treated with an oil-in-water emulsion containing a strongly-polar water-soluble molecule and a weakly-polar oil-soluble molecule which have a strong affinity for each other, resisted the onset of fretting to a greater extent than when a similarly coated steel was used in conjunction with a standard machine oil.

Solid lubricants, both metallic and non-metallic, have been used with considerable success in the alleviation of fretting corrosion. Thus, Godfrey and Bisson³⁰ have reported that when molybdenum disulphide was baked on to steel surfaces the onset of fretting damage was considerably delayed. Wright,⁸ however, is uncertain that molybdenum disulphide will have the required effect in every case since, under conditions of high load, there is the danger that the bonded film would fail at isolated spots and normal fretting would occur. A further disadvantage would appear to be the necessity to heat the coated steel at a temperature of about 350°C. in order to dry out the coating.

The principles by which thin films of various metals are used as lubricants have been discussed extensively by

Bowden and Tabor.¹⁶ In brief, metallic friction arises as a result of two processes, ploughing and shearing of asperities. Ignoring the former the frictional force F may be written simply as the product of A , the projected area of contact between the surfaces, and S , the stress required to shear the interacting asperities. Obviously to reduce the friction it is necessary to make both A and S as small as possible. With most metals this is not possible; where the metal shear strength is low, the contact area tends to be large, and vice versa. By coating a hard steel surface with a thin film of a metal with a low shear strength, however, it is possible to satisfy both requirements, since the hard steel backing to the low strength film limits the contact area. The low frictional characteristics of steel surfaces suitably coated with a metallic film leads to a considerable reduction in the damage occurring when oscillatory slip is present. In laboratory experiments, Gray and Jenny²⁷ have successfully demonstrated the ability of lead plating to eliminate fretting: indium has also been mentioned in this connection.

Selection of Material

Although the type of steel or other material selected depends largely on other considerations, it is worthwhile to bear in mind at the design stage the material characteristics which are likely to combat fretting if it should occur. In this connection, it has been observed that increasing the hardness of steels tends to reduce the damage resulting from fretting. If it is impossible to accept a high hardness in the body of a component it may be that some form of surface treatment would prove effective.

Avoiding Reactor Failures

FIVE generations of atomic reactor development leading to the new advanced gas-cooled reactor, with operating temperatures approaching 600° C., were described at a recent meeting of the Society of Non-Destructive Examination by Dr. M. Davis, who is personal assistant to the member for production of the United Kingdom Atomic Energy Authority, Sir Leonard Owen. In emphasizing that a shut-down resulting from either defective materials or faulty fabrication could seriously affect the overall cost of operation, Dr. Davis spoke of the careful inspection given to all components.

As a result of these inspection procedures, where material used for can manufacture was ultrasonically examined and every can weld was radiographed, it had been possible to reduce the total of faulty elements to about 4 per reactor per year, the number of fuel elements in the reactor being of the order of 10,000. There was hope of improving upon even this low figure. A very sensitive device, based on the detection of decaying gaseous fission products, had proved invaluable in giving early warning of a fuel element failure, so that the offending element could be discharged. The required degree of grain orientation in the all important uranium fuel was also being studied by ultrasonic methods. A novel method for the non-destructive examination of reactor channels was to be seen in the use of closed circuit television, whereby a camera complete with its own CO₂ cooling system could be lowered into a channel to inspect its contents and inside surface.

Dr. Davis pointed out that the pressure vessels used in nuclear engineering were the largest of their kind in the

Acknowledgments

Acknowledgment is made to those publications from which the diagrams have been reproduced, as indicated in the captions.

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world, and that their production had only been possible because of progress in welding and non-destructive examination. All plates were ultrasonically tested for freedom from lamination and each weld radiographically examined. Mega-voltage radiography was playing a more important part in the examination of the thicker welded vessels. Linear accelerators, recently developed in Britain, were being considered for use on site. The percentage sensitivity obtained when using a high activity cobalt source on a thick walled vessel was nearly as good as that given by a linear accelerator but the exposure time was much longer. To justify the greater cost of a linear accelerator, it would be important to exploit its short exposure time when it was employed for the site radiography of pressure vessels.

Incandescent Chemical Plant Division

For some years, the Incandescent Heat Co., Ltd., have manufactured process equipment to the designs of the Swenson Evaporator Co. (a division of the Whiting Corporation) of Harvey, Illinois. An agreement has now been reached by which Incandescent have exclusive selling and manufacturing rights in the United Kingdom for all Swenson products. To handle this work, a chemical plant division has been formed, headed by Mr. C. J. V. Denning. In addition to dealing with all enquiries for Swenson evaporators, crystallizers, filters and spray driers, the division will also handle sales of direct-fired heaters and combustors, air heaters and other specialised high temperature heat exchangers, high temperature furnaces, and other thermal equipment of interest to users of chemical process plant.

The Effect of Pickling and Anodising on the Fatigue Properties of 2L40 and D.T.D. 683 Aluminium Alloys

By J. M. Finney

(Structures Division, Aeronautical Research Laboratories, Melbourne, Australia.)

Because of certain advantages claimed for the caustic soda pickling of aluminium alloy forgings, an investigation was undertaken to assess the effect on fatigue properties of pickling and anodising 2L40 and D.T.D. 683 alloys, using both caustic-soda and A.I.D. standard solutions for the pickling operation. The result of these tests are presented here.

IN recent years the Australian aluminium industry has considered using a caustic soda solution to pickle aluminium alloy forgings in preference to other treatments that are used in connection with inspection and also with surface preparation prior to painting or anodising. This process is considered to be cheaper and more efficient; to enable cracks to be detected more readily; and to give a better surface finish than other pickles. However, the Aeronautical Inspection Directorate (A.I.D.) had prohibited the use of this pickle for aircraft materials,¹ evidently basing their objection on some Royal Aircraft Establishment (R.A.E.) work² reported in 1933, which suggested that it caused a reduction in fatigue strength.*

The use of caustic soda is also prohibited in the D.T.D. specification relating to anodising.³ Section 2.3—on 'Cleaning of Anodised Parts'—reads as follows: "When it is required to remove anodic films for re-anodising, this may be done, after degreasing, by one of the methods given in Appendix II. Caustic soda shall not be used."

A literature survey revealed that little additional information was available on this subject. It was therefore thought desirable to investigate the effect of pickling with both caustic soda and A.I.D. standard solutions on the fatigue properties of aluminium alloys, particularly in view of the facts that (a) caustic soda pickling is used in America^{4, 5}, apparently without deleterious effects; and (b) the R.A.E. work was concerned with a duralumin type (Al-Cu) alloy, and carried out before the introduction of the high strength Al-Zn-Mg alloys.

Two aluminium alloys of the types mentioned above, which are widely used in aircraft construction, were chosen for the investigation. One was a 4% copper alloy to specification 2L40B, and the other was a 5% zinc alloy to specification D.T.D. 683 3.

All pickled specimens were subsequently anodised, and comparisons were made with the fatigue strength of untreated specimens. One batch of specimens from each material was also anodised without prior pickling. The pickles used were:—

- sulphuric-hydrofluoric acids,
- orthophosphoric-hydrofluoric acids,
- sulphuric acid—potassium fluoride,
- sulphuric acid—sodium fluoride,
- caustic soda (10%), and
- caustic soda (20%).

Details of these pickles are given in an Appendix.

* A more recent Instruction, however, allows restricted use⁶

TABLE I.—TENSILE PROPERTIES OF TEST MATERIALS

Material	0.1% Proof Stress (lb./sq. in.)	0.2% Proof Stress (lb./sq. in.)	Ultimate Tensile Strength (lb./sq. in.)	Elong. on 2 in. (%)	Brinell Hardness Number*
2L40 (Average of 13 specimens) . .	63,400	63,800	71,000	8.4	136
D.T.D. 683/3 (Average of 16 specimens) . .	82,700	83,700	89,200	9.2	165

* Made on approximately 25% of the fatigue specimens, using a Vickers hardness testing machine employing a 2 mm. ball and a load of 40 kg.

Material and Specimens

The 2L40B material was taken from one batch and supplied in thirteen lengths of $\frac{9}{16}$ in. diameter extruded bar. The higher strength D.T.D. 683/3 alloy was also taken from one batch and supplied in sixteen lengths of $\frac{13}{16}$ in. diameter extruded bar. Chemical analyses were performed on samples from each bar to check conformity with specification. The first specimen in each bar was made into a standard tensile specimen, the test results being given in Table I. The remainder of each bar was machined into unnotched rotating cantilever fatigue specimens (Fig. 1) using a profiling lathe, the parallel portion and test contour being machined in one setting. The speeds and feeds for the various machining operations were as follows:—

Roughing Cuts : speed 900 r.p.m., feed 0.0045 in./rev.
Finishing Cut : speed 1,800 r.p.m., feed 0.0022 in./rev.
Depth of Cut : approx. 0.010 in.

Experimental Procedure

Pickling and Anodising

The pickling operation was performed using 3 litres of solution, all the specimens necessary for a complete S-N curve being mounted in a jig, degreased, and pickled in one operation. During this operation they were constantly agitated by hand.

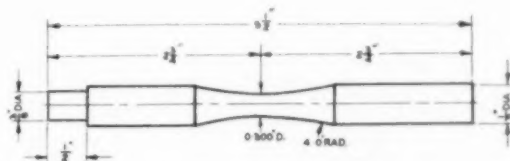


Fig. 1.—Fatigue specimen

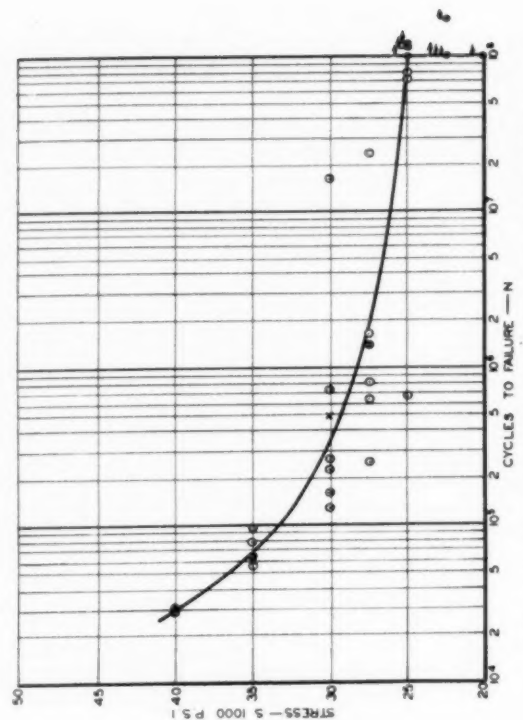
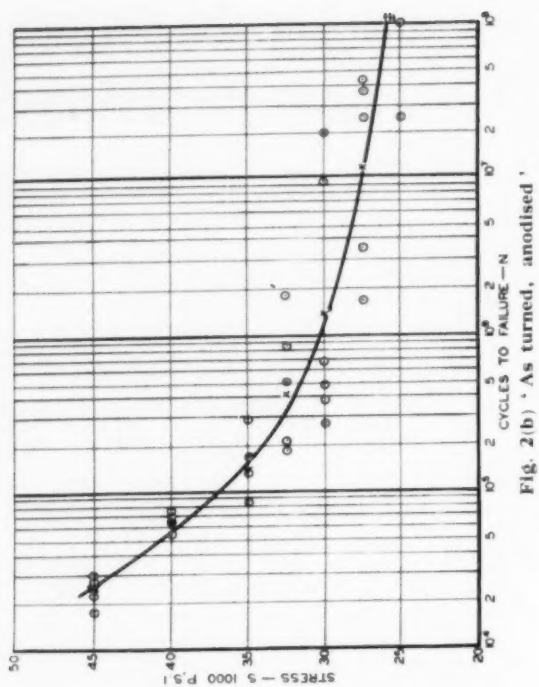
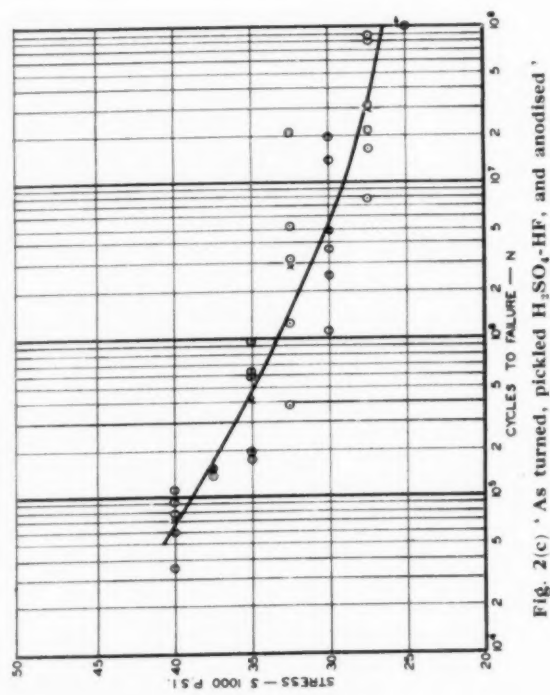
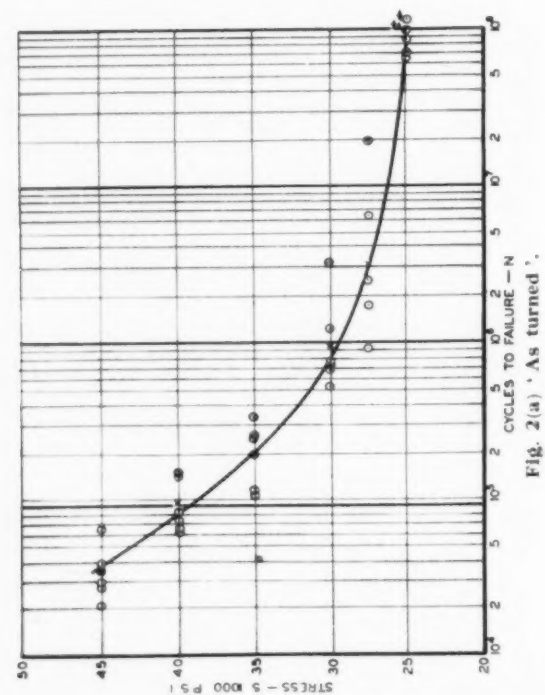


Fig. 2. S-N curves for 2L40 aluminium alloy.

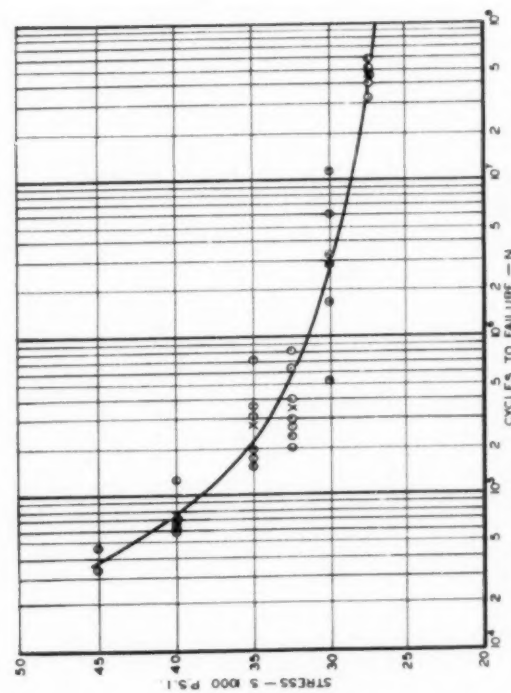


Fig. 2 (e) 'As turned, pickled H_2SO_4 -KF, and anodized'

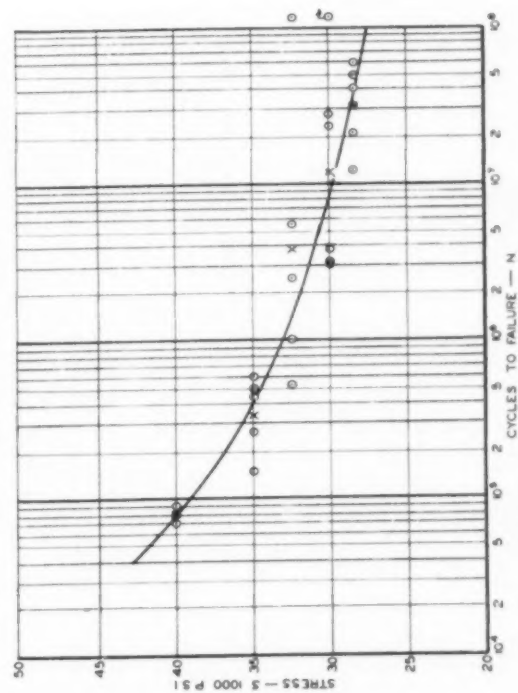


Fig. 2 (g) 'As turned, pickled 10% NaOH, and anodized'

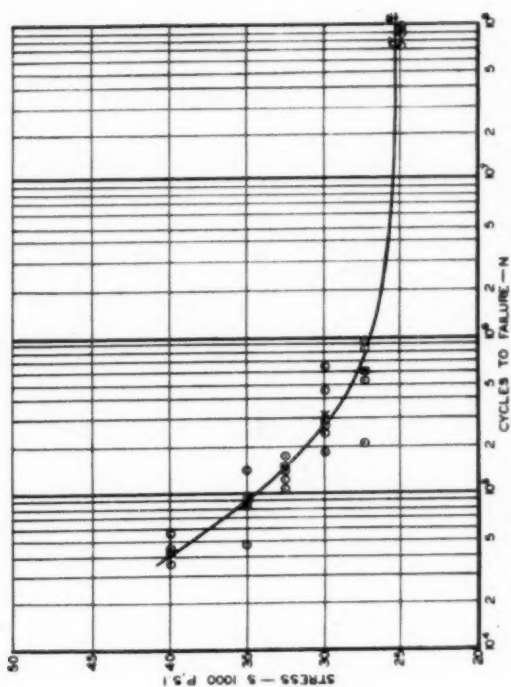


Fig. 2 (f) 'As turned, pickled H_2SO_4 -NaF, and anodized'

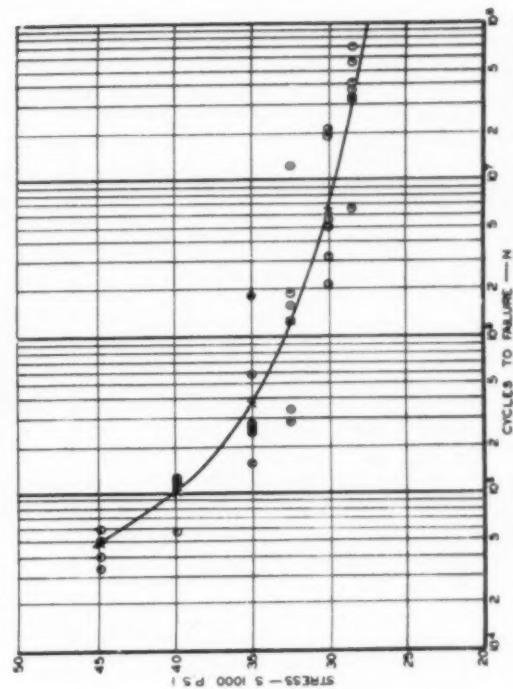


Fig. 2 (h) 'As turned, pickled 20% NaOH, and anodized'

Fig. 2. —S-N curves for 2L40 aluminium alloy.

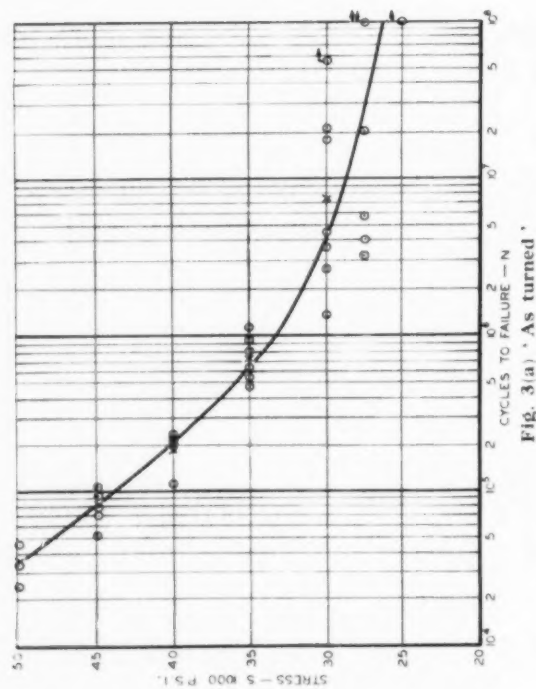


Fig. 3(a) 'As turned'

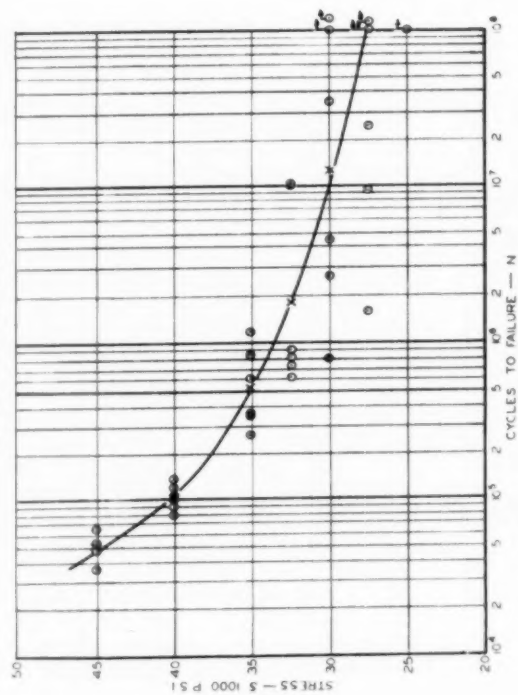


Fig. 3(c) 'As turned, pickled H_2SO_4 -HF, and anodised'

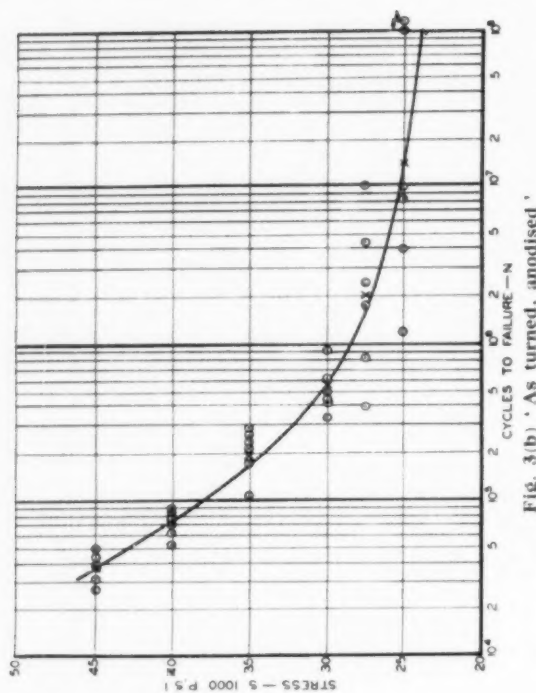


Fig. 3(b) 'As turned, anodised'

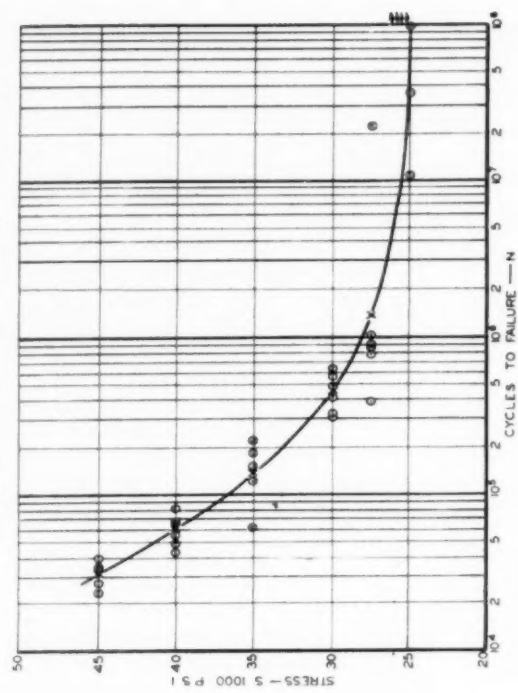


Fig. 3(d) 'As turned, pickled H_2PO_4 -HF, and anodised'

Fig. 3. —S-N curves for D.T.D. 683 aluminium alloy.

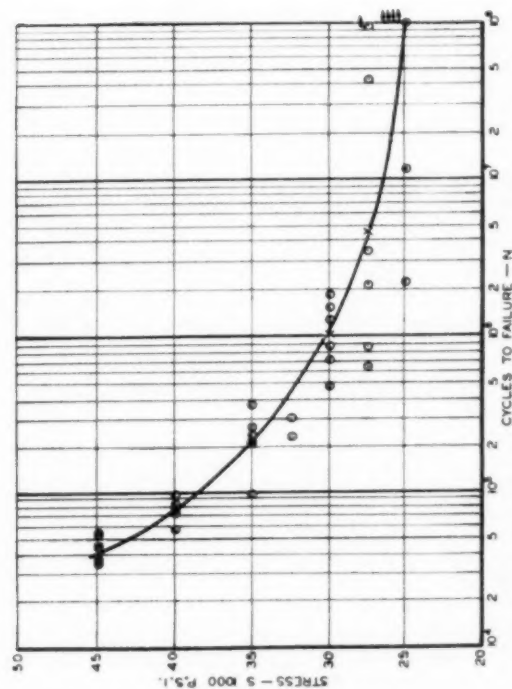


Fig. 3 (e) 'As turned, pickled H_2SO_4 -KF, and anodised'

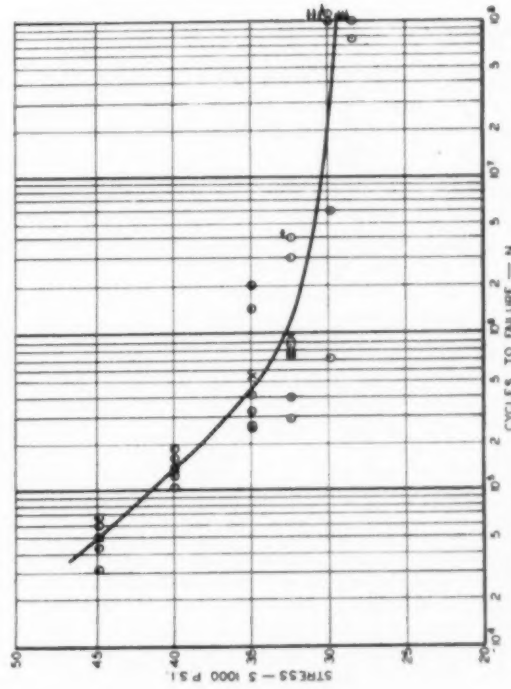


Fig. 3 (g) 'As turned, pickled 10% NaOH, and anodised'

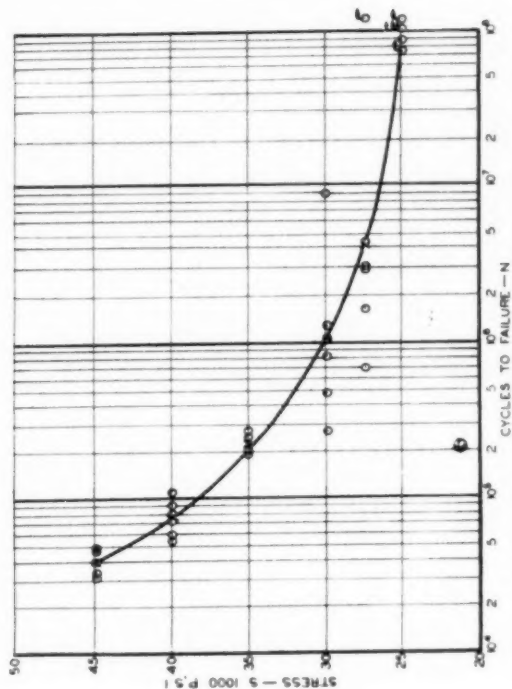


Fig. 3 (f) 'As turned, pickled H_2SO_4 -NaF, and anodised'

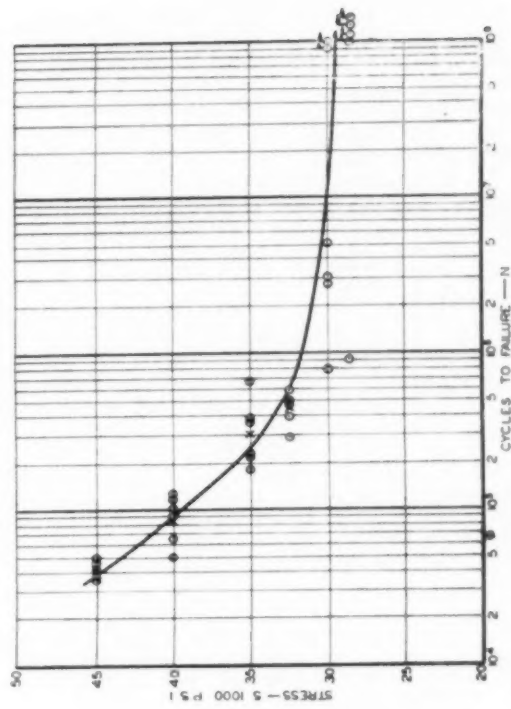


Fig. 3 (h) 'As turned, pickled 20% NaOH, and anodised'

Fig. 3. S-N curves for D.T.D. 683 aluminium alloy.

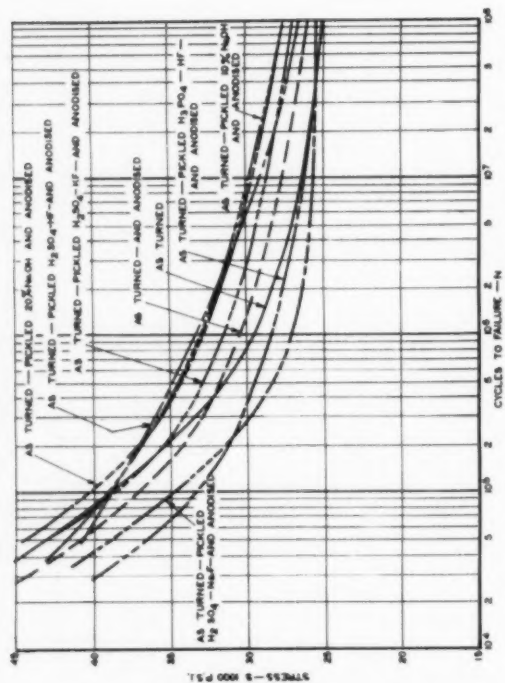


Fig. 4.—S-N curves for 2L40 aluminum alloy.

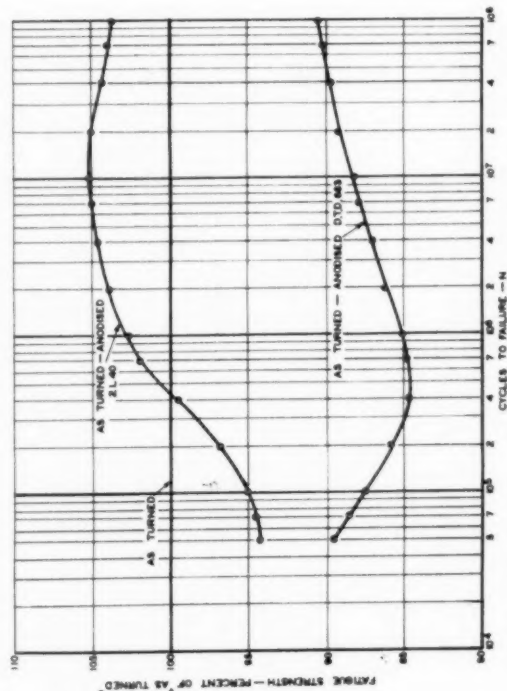


Fig. 6.—Effect of anodising on 2L40 and D.T.D. 683 aluminium alloys.

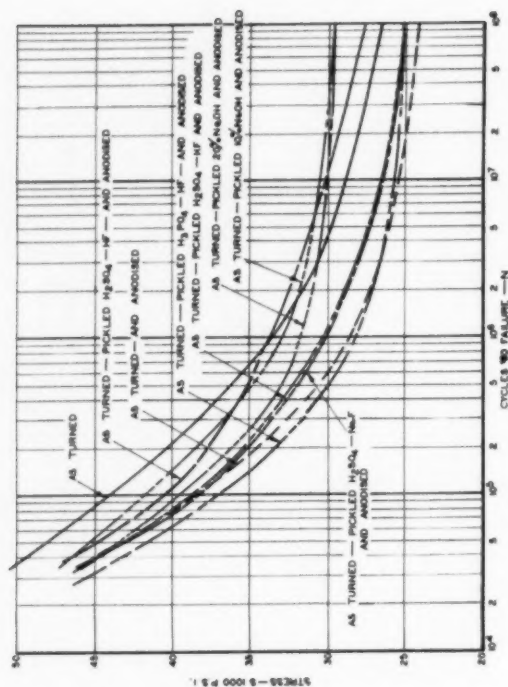


Fig. 5.—S-N curves for D.T.D. 683 aluminium alloy.

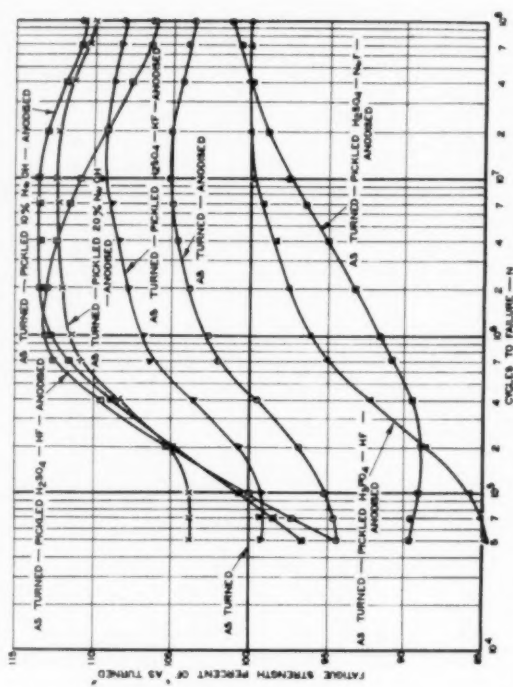


Fig. 7.—Comparison of anodising and pickling processes on the fatigue strength of 2L40 aluminium alloy.

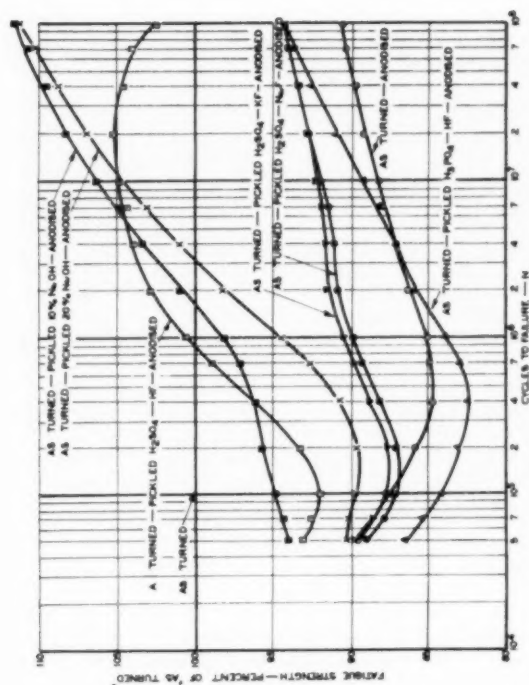


Fig. 8.—Comparison of anodising and pickling processes on the fatigue strength of D.T.D. 683 aluminium alloy

Anodising was performed in a chromic acid bath to specification D.T.D. 910C. The lapse of time between pickling and anodising, and between anodising and the commencement of testing, was arbitrarily standardised at 1½–2 hours in both cases.

Fatigue Tests

All fatigue tests were performed in the Aeronautical Research Laboratories rotating cantilever fatigue machines operating at 12,000 c./m. In most cases, each S-N curve was obtained by testing six specimens at each of six stress levels.

To eliminate any preferential effects due to variations in bar stock or between testing machines, the following procedure was adopted for each S-N curve.

Immediately after machining, the specimens were sorted so that each S-N curve was obtained by using approximately the same number of specimens from any one bar, no two specimens from any bar being tested at each stress level. Three machines were employed for the investigation, each machine being used to test two specimens at each stress level for every S-N curve.

Surface Profile Assessment and Depth of the Anodised Layer

Both the surface profile and the thickness of the anodised layer of each batch of specimens were examined, using the chord sectioning technique developed by Ryan.⁷ To eliminate variations in magnification due to the change in diameter in the test section of the specimen, samples for chord sectioning were taken from the parallel portion of the specimen. It was assumed that the surface profile was the same at both places, as the specimens were machined in one operation in a profiling lathe. Surface profile measurements using a Brush Surface Analyser confirmed this assumption.

Results of Tests

Fatigue Tests

The results of fatigue tests on both alloys treated by the various processes outlined in the Appendix, are presented as S-N curves in Figs. 2 and 3. The single curve shown in each of the figures is based on the logarithmic mean of the cycles to failure. These curves are replotted, without the test points, in Figs. 4 and 5 for the 2L40 and D.T.D. 683 alloys, respectively. To facilitate comparison, the relative fatigue strengths based on the 'as turned' endurance curve have been plotted for each alloy—2L40 in Fig. 7 and D.T.D. 683 in Fig. 8.

Surface Profile Assessment and Depth of the Anodised Layer

Typical examples of specimens prepared by the chord sectioning technique are shown in Fig. 9. In the photograph of the anodised specimen, the white portion is the base metal, while the black line above the metal is the junction of the anodised surface and the "Bostik" coating. In comparing these photographs, the difference in vertical magnification must be considered.

The average thickness of the anodised layer on any specimen was also obtained using the chord sectioning method. For both alloys the values obtained from different specimens ranged from 0.00013 in. to 0.00018 in., the average being 0.00014 in. The accuracy of any individual result was estimated to be within 0.000015 in. The average value of 0.00014 in. is just over one-half the thickness which is quoted for the chromic acid process,⁸ but different methods of measurement in the two cases may be a reconciling factor.

Discussion of Results

Effects of Pickling and subsequent Anodising on Fatigue Strength

In the case of aluminium alloys, pickling is employed both to facilitate inspection and to cleanse components before anodising or painting. This report deals with tests on specimens which were anodised after pickling, thus simulating one set of practical conditions. All results discussed herein relating to the effects of pickling apply to the combined effect of pickling and anodising, fatigue test results on pickled specimens being compared with 'as turned' results for each alloy.

The results will be discussed under two headings, namely: (i) standard A.I.D. (acid) pickles, and (ii) caustic soda pickles.

(i) *Standard A.I.D. (Acid) Pickles*—The influence of these acid pickles on the fatigue behaviour of the two alloys is shown in Figs. 4 and 5, or 7 and 8. These show that the relative effects of the different pickles depend upon the endurance chosen, thus making generalisations difficult. They also show that the effect of a given pickle differs with each alloy.

At short endurance—of the order of 5×10^4 cycles—all pickles decreased the fatigue strength of both alloys. For the 2L40 aluminium alloy, all acid pickles, except the H_3PO_4 -HF pickle, increased the fatigue strength at 10^6 cycles. The H_3PO_4 -HF pickle had no apparent effect at this endurance. For the D.T.D. 683 aluminium alloy, only the H_2SO_4 -HF pickle gave increased fatigue strength at 10^6 cycles. An interesting feature of the H_2SO_4 -KF and H_2SO_4 -NaF pickles is that with the D.T.D. 683 alloy

their effects were practically identical at all endurance, whereas with the 2L40 alloy their quantitative effects differed considerably at all endurance.

Only meagre information exists on the effects of some of these pickles (without anodising) on fatigue properties. Sutton and Peake⁹ reported a decrease of approximately 7% at 10^7 cycles in rotating cantilever fatigue properties of duralumin (4.25% Cu, 0.93% Mg), when pickled in a solution consisting of 10% H_2SO_4 and 1% NaF. Pickling the same alloy in 20% H_3PO_4 + 0.5% HF reduced the fatigue strength by approximately 9%. These results should, however, be treated with caution, as only about six specimens were tested to obtain each S-N curve, and thus the small apparent difference in fatigue strength may be insignificant.

Müller¹⁰ reported no difference in the flexural fatigue properties of Avional sheet (3.5-5.0% Cu, 0.2-1.5% Mg, 0.2-1.5% Mn, 0.1-0.5% Si) when pickled in 10% H_2SO_4 + 1% NaF.

(ii) *Caustic Soda Pickles*—In 1933, Sutton and Taylor² investigated the effect of pickling (without subsequent anodising) with a 10% caustic soda solution on the rotating cantilever fatigue properties of duralumin (B.S. 3L1). Briefly, their conclusions were that: (a) this procedure resulted in a decrease of fatigue strength of approximately 30% at 10^7 cycles; (b) after immersing pickled specimens in boiling water, the reduction in fatigue strength was only 10%; and (c) normal properties were restored after removing a layer 0.0025 in. thick by machining. Whilst it is recognised that these investigators found a considerable reduction in fatigue properties due to the caustic soda pickle, nevertheless the magnitude of the reductions quoted are open to doubt, as again only approximately six specimens were used to determine each S-N curve, and the S-N curves were apparently arbitrarily drawn as two straight lines.

Müller¹⁰ reported tests on Avional (Al-Cu-Mg alloy) in which bar specimens were pickled for 10 minutes in 10% NaOH. The resulting reduction in fatigue strength was reported to be approximately 12%. When pickled for 40 minutes in the same solution, the reduction was increased to about 25%. The same author also reported tests on Avional sheet, pickling in a 10% NaOH solution for 30 seconds and 8 minutes, respectively, but in contrast to his previous results, in both cases there was no change in fatigue strength (at 10^7 cycles).

A limited number of sheet bending fatigue tests of L71 (Al-Cu alloy) have been reported¹¹ using specimens either milled or severely etched in 10% NaOH at 80°C. No difference in fatigue strength (at 2×10^5 cycles) was found between specimens prepared by these processes.

It should again be emphasised, however, that all the results quoted above on the effect of caustic soda pickling were derived from tests on pickled but unanodised specimens, and hence are not strictly comparable with the results reported herein.

From Figs. 7 and 8 it may be seen that, for both alloys, at endurance greater than 10^7 cycles, caustic soda pickling gives the highest fatigue properties of all the conditions investigated. It must, however, be pointed out that, in the case of the D.T.D. 683 alloy, the caustic soda and anodising treatments have decreased the fatigue properties at short endurance, but nevertheless they still result in fatigue properties superior to most of those resulting from the acid pickles. It should also be noted that the 20% NaOH pickle results in properties

slightly lower than those obtained with the 10% NaOH pickle, thus suggesting that there may be an optimum NaOH concentration for a particular time and temperature of pickling.

Effect of Anodising on Fatigue Strength

Fig. 6 shows that chromic acid anodising (without pickling) affected the fatigue properties of each alloy in different ways. The process decreased the fatigue strength of the D.T.D. 683 alloy at all endurance, but increased that of the 2L40 alloy at endurance greater than 5×10^5 cycles. These results are in contrast to those of Stickley and Howell¹² who suggest that the type of alloy has no effect, but that the thickness of the anodic coating is the governing factor. As reported above the thickness of the anodic coating on specimens tested in the current investigation is in the order of 0.00013-0.00018 in. for both alloys. Although there have been a number of papers published on the effect of anodising on fatigue strength, it is not proposed to discuss further this aspect in the present paper.

Surface Profile and Residual Stresses

Pearson,¹³ who investigated the effects of various pickling reagents on the surface profile of Alcan 26S-T (Al-Cu alloy) and Alcan 75S-T (Al-Zn alloy), concluded that "The surface produced by the sodium hydroxide treatment is somewhat different in nature from that produced by the other solutions (H_2SO_4 -NaF and H_2SO_4 -KF). The pits are less angular and the ratio of pit width to pit depth is much greater than in the case of the pits resulting from the sulphuric acid solutions." He also concluded from his results that "from the standpoint of fatigue, sodium hydroxide is likely to be as satisfactory as the acid solutions for these two alloys." This conclusion is in agreement with the present results, although there is little evidence from the chord sections to indicate that differences in fatigue properties due to the various treatments are related to differences in surface profile.

As the volume of the anodic layer is greater than that of the aluminium from which it is formed (the anodic film is composed of hydrated aluminium oxide¹⁴), it is possible that the anodising introduced residual stresses into the surface layers of the specimens. If this is so, these stresses may modify the specimen fatigue behaviour although one might assume that their effect would be identical in each case.

During the machining of the specimens a certain amount of work hardening and residual surface stress are introduced. The quantity of material removed by the different pickles was not investigated, but differences may have been sufficient to alter relatively the characteristics of the surface layers of the material.

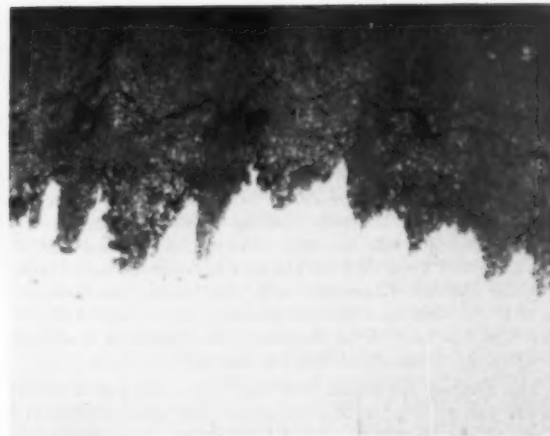
Conclusions

(1) Tests showed that the effects of various pickling treatments followed by anodising, on the fatigue properties of 2L40 and D.T.D. 683 aluminium alloys, were as follows:—

(i) At endurance greater than 10^7 cycles, pickling in caustic soda resulted in fatigue strengths superior to those obtained by using any approved A.I.D. acid pickle. At endurance less than 10^7 cycles, the caustic soda pickles resulted in fatigue strengths equal to those obtained from using the best acid pickle.



(a) Specimen 'as turned': vertical magnification approx. 4,600; horizontal magnification 250.



(b) Specimen 'as turned and anodised': vertical magnification approx. 5,200; horizontal magnification 250.

Fig. 9.—Chord sections of D.T.D. 683 specimens.

(ii) In the case of acid pickles, the best fatigue properties for both alloys were obtained after treating with the H_2SO_4 -HF solution.

(iii) In general, pickling and anodising caused a much greater reduction in strength in the case of the D.T.D. 683 than for the 2L40 alloy.

(2) Compared with 'as turned' specimens, anodising by the chromic acid process, without prior pickling, affected the fatigue behaviour of the two alloys investigated in a different manner. For the 2L40 alloy the general effect was beneficial, but for the D.T.D. 683 alloy it reduced the fatigue strength by as much as 15%.

Acknowledgments

This paper is published by permission of the Chief Scientist, Department of Supply, Australia. Acknowledgment is due to Messrs. F. G. Lewis and P. Scott-Young for advice and metallographic work respectively; also to Mr. J. Y. Mann for initial help, to Mr. N. E. Ryan for the chord sections, and to Mr. W. W. Johnstone for his supervision and suggestions throughout the project. The assistance of the Government Aircraft Factories, Fishermen's Bend, Melbourne, in performing the anodising process, is also gratefully acknowledged.

Appendix

Details of Pickling Processes

The six pickles and procedures applied to each alloy are detailed below. The first four were carried out according to A.I.D. Inspection Instruction M310¹. The fifth and sixth were identical with the procedure used by Sutton and Taylor in 1933,² except that in the latter case the concentration of the NaOH solution was doubled.

- (1) *Sulphuric Acid—Hydrofluoric Acid Pickle* (H_2SO_4 -HF)
Sulphuric Acid (10% acid) 99% by volume
Hydrofluoric Acid (commercial 50-60%) 1% by volume
Specimens warmed in hot water and then pickled at room temperature for 5 minutes.
- (2) *Orthophosphoric Acid—Hydrofluoric Acid Pickle* (H_3PO_4 -HF)
Orthophosphoric Acid (S.G. 1.5) 20% by volume
Hydrofluoric Acid (commercial) 0.5% by volume
Water remainder
Specimens pickled at room temperature for 5 minutes.

- (3) *Sulphuric Acid—Potassium Fluoride Pickle* (H_2SO_4 -KF)
Sulphuric Acid 10% by volume
Potassium Fluoride 4% by weight
Water remainder
Specimens pickled at room temperature for 5 minutes.
- (4) *Sulphuric Acid—Sodium Fluoride Pickle* (H_2SO_4 -NaF)
Sulphuric Acid 10.5% by volume
Sodium Fluoride 1.125% by weight
Water remainder
Specimens pickled at room temperature for 10 minutes.
- (5) *Caustic Soda A* (NaOH)
Caustic Soda 10% by weight in water
Specimens pickled at 65°C for 2½ minutes.
- (6) *Caustic Soda B* (NaOH)
Caustic Soda 20% by weight in water
Specimens pickled at 65°C for 2½ minutes.

The final operation of each pickling procedure was to immerse the specimens in a cold 50% nitric acid solution for 1 minute.

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BULK deliveries of Phosbrite 159 chemical polishing solution are now being made to Haynes, Ford and Elliott, Ltd., the Birmingham metal finishing firm, by Albright and Wilson (Mfg.), Ltd. Haynes, Ford and Elliott have installed a 1,000 gal. storage tank to take the deliveries, which are made by 6 ton tanker.

An Experiment in Research

Progress at Pioneer British Sponsored Research Institute

AT their opening in June, Lord Hailsham remarked that the Warren Spring Laboratories of the D.S.I.R., which could take on contract research for industry, were themselves an experiment in research, but the Fulmer Research Institute, which was founded in 1947, is also an experiment along these lines and the same may be said of the Soudes Place Research Institute at Dorking, which was founded in 1948.

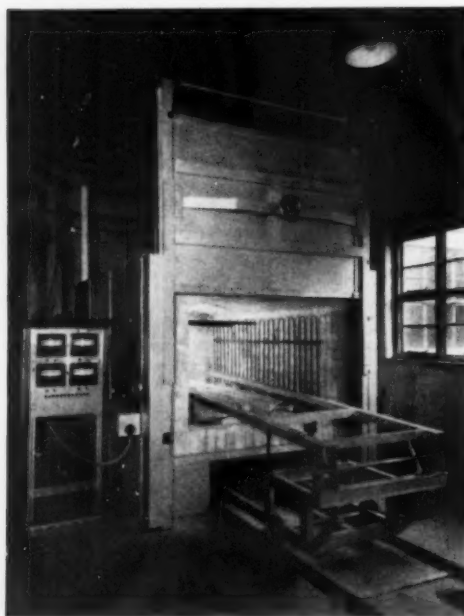
The Fulmer Research Institute was set up as a result of private enterprise and initiative and differs from the research associations and Government laboratories in that it receives no annual grant or income except in payment for services rendered. Since it is under no direct obligation to the taxpayer, it is entirely free from Government control, and no particular section of industry has any rights or priority in research results or facilities, except in so far as these have been paid for directly. This is a feature that often appeals to industrial sponsors, to whom commercial security is important, and who wish to have complete control of the research work for which they pay. Co-operatively and nationally financed research is carried out by research associations and Government laboratories and, although a certain amount of confidential or sponsored research is now being undertaken by them, their activities must be subject to control and scrutiny by the bodies responsible for the overall financing of the laboratory. The Fulmer Research Institute, therefore, fulfils a very necessary function in providing research facilities for progressive

organisations that have no adequate facilities themselves, but who wish to keep both their problems and aims confidential. The Institute is also useful to large organisations with adequate facilities and finance, who are either too busy to carry out certain investigations themselves, or who would like a fresh approach to a problem, or who approach Fulmer because of its pre-eminence or specialised techniques in certain fields.

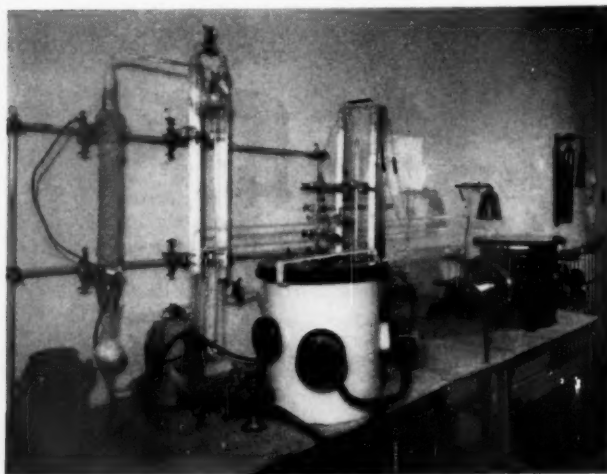
The Fulmer experiment in research differs from Warren Spring in that it has been financed on a very much more modest scale, and the steady expansion of the Institute has been achieved solely from profits ploughed back. This has resulted in the strictest economy being observed in buildings and fitments, and the total expenditure on all land and buildings at Fulmer, including fitments and services, has been £72,000, representing approximately £650 per person employed, or £2,000 per graduate. The comparable figures for the Warren Spring Laboratories are approximately £2,000 per person employed and about £6,000 per graduate, which is still low in comparison with other industrial laboratories built with an eye to prestige. The financial requirements of research management are affected to quite a considerable extent by whether the money is available as a grant, or whether it has to be either borrowed or earned.

The results of the Fulmer experiment in research which have emerged so far are:—

- (1.) It is possible to make research pay for itself and Government grants and endowments are not essential, although they give a decided advantage and it may be difficult for the research organisation without such aid to compete. However, the Fulmer Research Institute has always had a small but steady excess of income over expenditure,



Enamelling furnace.



Vacuum-argon arc dry box for handling reactive materials.

which has enabled a moderate programme of expansion to be undertaken.

- (2). It is possible to do first-class scientific work on a sponsored basis. As evidence of this we would point to the scientific papers which have been published, which are, of course, only those for which the sponsor's permission to publish has been obtained.
- (3). There is a demand from publicly owned Corporations and Government Departments for independent research work. At present, 42% of the Institute's income comes from the Atomic Energy Authority and 25% from the Ministry of Supply and other Government Departments.
- (4). There is a demand from British industry for sponsored work, but about half of this demand comes from small firms who are more interested in test and consulting work than basic research. Although British industry at present only contributes 18% of the Institute's income, this amounts to £27,000, which is greater than the total income of the Institute in the first year of operation and it is growing steadily. About £10,000 of this is represented by test work and consulting services, and there is a tendency for this work to increase both in percentage and total.
- (5). Compared with British industry, there is a greater and more spontaneous demand for research work from industry in the United States and Canada, and 15% of the Institute's income is from these sources. Practically none of this demand from overseas is for test or consulting services, and the proportion of long term or basic research work which is being carried out for the U.S.A. and Canada is higher than that of similar work for British industry.

In considering these results it must be borne in mind that the Institute has at all times been working at or near capacity, and no special effort has been made to obtain one sort of sponsorship rather than another. It is none the less hoped that in the future British industry will demand a greater share in the use of the research facilities of the Institute.

In the following pages a brief account is given of the equipment available—much of it for the carrying out of specialised techniques—and of some of the fields in which research is being, or has been, undertaken. It will be realised of course that little or no information can be given in some cases, owing to the confidential nature of the problem.

Equipment and Specialised Techniques

X-Ray Crystallography—Extensive facilities are available for X-ray diffraction work, and six X-ray generators are in use. Conventional crystallographic methods are supplemented by specialised techniques for studying reactive metals. A high temperature X-ray camera enables readily oxidised materials such as zirconium and titanium alloys to be studied at temperatures up to 1,000°C. in a vacuum of 10^{-7} mm. of mercury. The structure of liquid metals at elevated temperatures is being studied with a specially designed Geiger counter X-ray diffractometer, and single crystal methods using monochromatic radiation are employed for studying the mechanism of age-hardening and phase transformations.

Electron Microscopy—A Metropolitan-Vickers EM3A

electron microscope is used for reflection and transmission (thin film) microscopy and electron diffraction work. In recent researches, the microscope has been used to investigate the build-up of deposits in oil burning furnaces, the fundamental nature of slip lines, and surface oxidation studies. Other uses include examination of paint pigments and contaminated catalysts.

Metallography—In addition to a Bausch & Lomb Metallograph and a Reichert Metallurgical Microscope, there are several bench microscopes, one of which is equipped for phase contrast microscopy and micro-hardness testing. Conventional heat treatment equipment is supplemented by a vacuum quenching apparatus and a high temperature vacuum furnace which can operate at temperatures up to 2,000°C. with a vacuum better than 10^{-5} mm. of mercury. A special dilatometer incorporating a differential transformer has been constructed for studying isothermal transformations and martensitic transformations on rapid quenching. The apparatus was designed for studying uranium alloys but can be used for studying transformations in most metals.

Physical Chemistry—Specialised techniques in the field of physical chemistry include accurate measurements of vapour and reaction pressure, using the capillary vessel method developed in the Institute, in addition to all standard methods. These have been used to study the stability of radicals at high temperatures and to establish activity data for various metallic systems. Accurate calorimetry has established the heats of formation of various compounds, in particular metal halides and intermetallic compounds, and new values for the heats of formation of a large number of important compounds in these categories have been established and published. The kinetics and equilibria of various reactions of interest in the fields of nuclear power and extraction metallurgy have been studied in apparatus requiring highly efficient vacuum techniques.

Melting and Casting—Apart from the normal oil and gas-fired furnaces for melting, induction furnaces have been adapted for melting and casting in special atmospheres or *in vacuo* and for zone refining as well as for open melting. Most reactive alloys for experimental work are prepared in argon arc melting furnaces with water-cooled copper hearths. One of these is equipped with a retractable hearth from which small billets suitable for extrusion can be made. An apparatus has been constructed for melting by electron bombardment, and is used for zone refining and growing single crystals.

Metal Working—A forging hammer and a small rolling mill are available. From the latter either sheet or rod can be produced, and techniques for sheath rolling reactive alloys are well established. There is also a 60-ton press for use with powder metallurgical techniques, and a miniature extrusion press. Arrangements have been made outside the laboratories for the extrusion of highly reactive and refractory materials, some of which have been sheathed and extruded using glass lubrication. This technique has proved particularly valuable in the investigation of chromium and chromium base alloys.

Refractories—Special techniques for the preparation of refractories for dealing with highly reactive metals have been developed. A kiln is available for firing refractories at temperatures up to 2,000°C., and ball and roller mills allow the preparation of finely divided material for slip casting or spraying. A diamond cut-off wheel provides

for accurate shaping and cutting of thin slices of hard brittle substances.

Mechanical Testing—The engineering laboratory is equipped to carry out all normal mechanical tests. In addition to impact and hardness machines, a Denison 50 ton universal testing machine with ancillary equipment for high and low temperature testing is in use. Wöhler, Haigh, "slipping clutch" and Rolls Royce machines are available for fatigue testing, and equipment has been specially designed for high temperature fatigue testing of sheet. Interesting information about the initiation of fatigue cracks is being obtained by special replica techniques.

Conventional creep testing apparatus is supplemented by facilities for carrying out creep in compression and in special protective atmospheres. This has proved essential in studies of compression creep of uranium and other highly reactive metals. Both static and dynamic strain gauge measurements have been made on structures under load. This work has been done in the field as well as in the laboratory.

Chemical and Spectrographic Analysis—Conventional polarographic, absorptiometric and spectrographic methods are available for the analysis of metallurgical and inorganic materials. The analytical equipment has recently been supplemented by the acquisition of a cathode-ray polarograph. The co-ordinated effort in using a combination of appropriate techniques has enabled new methods to be developed for ore analysis, and for trace element determination in phosphors, pharmaceutical products, graphites, etc.

Corrosion—Laboratory corrosion test apparatus includes salt spray, humidity and SO_2 cabinets and high accuracy quartz spring balances for studying oxidation in special atmospheres by weight changes. For atmospheric corrosion the Institute has exposure sites in industrial, marine and rural environments. A novel technique has been developed for non-destructive assessment of corrosion damage in terms of loss of mechanical strength, enabling a corrosion/time curve to be found on a single specimen. Electronic equipment such as valve voltmeters, a potentiostat and a constant current device are used in studying cathodic protection.

Electrodeposition—Four separate rectifiers deliver direct current supplies up to 250 A. and 60 V., enabling experimental electrodeposition to be carried out on a scale ranging from beakers to full pilot plant. These facilities are used extensively for the preparation of very pure chromium: a production rate of 25 g./hr. is possible. Work has also been carried out on the electrodeposition of manganese, and on the anodising and dyeing of aluminium and its alloys.

Fields of Investigation

Thermochemistry—Mention has been made under the heading "Physical Chemistry" of some of the techniques employed for making thermodynamic measurements. Determination of heats of formation is vital to the assessment of the feasibility and yield of various metallurgical processes, including extraction, fluxing, alloying and coating. Values have already been obtained for the following compounds: aluminium and magnesium fluorides; cryolite; titanium tetrachloride, tetrabromide and tetrafluoride; sulphur hexafluoride; zirconium, hafnium and vanadium tetrachlorides; and

niobium and tantalum pentachlorides and pentabromides. The high heats of formation of fluorides make these compounds potentially important as rocket propellants.

In physical metallurgy, as well as in extraction metallurgy, free energies and heats of alloy formation are important parameters of intermetallic systems, and the following systems have been investigated: iron-aluminium-silicon arc furnace alloys, iron-chromium-carbon, aluminium-manganese, iron-titanium, iron-aluminium, uranium-bismuth, thorium bismuth, and uranium-silicon. In this work, each system requires a special approach, and in the case of the highly reactive uranium and thorium alloys novel techniques are used for preparation and handling.

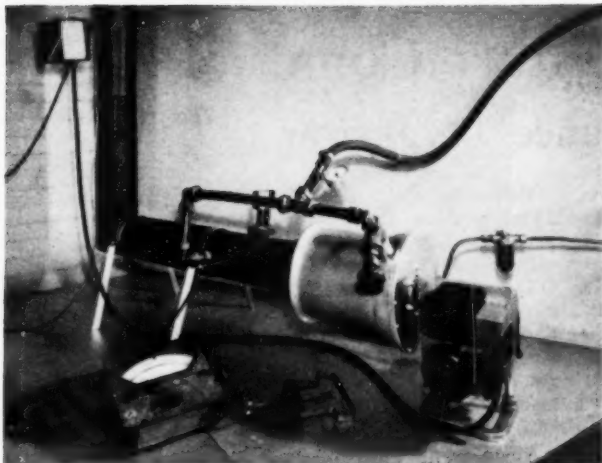
Extraction Metallurgy—One of the first investigations carried out at the Institute arose from the development by Dr. Gross of the well known aluminium catalytic distillation process. This process depends upon the reversible reaction between aluminium trichloride and an aluminium-bearing material to form a volatile monochloride, which subsequently decomposes into aluminium and aluminium trichloride. The reaction is applicable to the extraction of aluminium from alloys produced by direct thermal reduction of bauxite, or other suitable aluminium-bearing minerals, in an arc furnace, or to purification of scrap. Aluminium of high purity has been produced by this method. Extensive development has been carried out, and laboratory work is continuing at the Institute.

A similar approach has been used in the development of a titanium extraction process in which titanium tetrachloride is passed over ferro-titanium or titanium scrap to form titanium di- and tri- chlorides, which are subsequently decomposed to form relatively pure titanium and further tetrachloride for recirculation. The process has been developed on a laboratory scale and can be extended to the pilot plant stage. A related method has been applied to the purification of beryllium, with promising results.

Uranium Alloys and Compounds—Recent work in uranium metallurgy has included extensive phase diagram and transformation studies on binary and ternary alloys. X-ray crystallography is being used to elucidate the structure of uranium chelate complexes, and various uranium and uranium alloy phases. Creep tests on uranium and its alloys have been carried out in compression. Other uranium work in progress has been referred to under the heading "Thermochemistry".

Zirconium and Zirconium Alloys—A study is being made of the w -phase in zirconium alloys and the binary systems with niobium, molybdenum, uranium and chromium, have been examined so far. The w -phase acts as an embrittling agent during heat treatment of zirconium alloys for use in nuclear reactors. The high temperature X-ray diffraction camera, previously mentioned under "X-ray Crystallography" and an apparatus in which specimens can be quenched in helium at 8,000° C./second are used in this work. The mechanism of oxidation of zirconium and zirconium alloys in CO_2 is also being studied.

Age Hardening—The original metallurgical work on age hardening of aluminium alloys supervised by Dr. Hardy, Beilby Award winner, is continuing and this, together with the X-ray techniques developed at the Institute for studying precipitation processes, is well



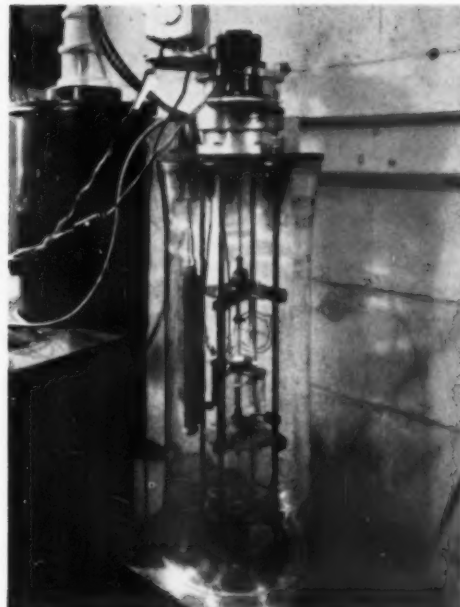
Oil burning rig used in work on the build up of furnace deposits.

known internationally. Trace elements have been shown to exercise a profound effect on the ageing behaviour in some systems, and the influence of small quantities of cadmium in accelerating the ageing of aluminium-copper alloys led to the development of the aluminium-copper-cadmium alloys, which, while free from room temperature ageing after solution treatment, can be aged at slightly elevated temperatures to give properties approaching those of the duralumin type alloys. These alloys have excellent pressing properties and machining stability, and work is in progress to assess their suitability for structural work. Trace element effects have also been found in magnesium-copper and silver-base alloys.

Aluminium Alloys—In addition to the work on age hardening aluminium alloys described above, various other researches are in progress, including a study of the mechanisms of layer corrosion and stress corrosion in structural aluminium alloys. The influence of thickness of anodised coatings on the corrosion resistance of various aluminium alloys is also being studied, and an investigation is in progress on the influence of metallic coatings anodic to the basis metal on the corrosion fatigue of high strength alloys. An aluminium-tin bearing alloy containing up to 30% tin, developed in conjunction with the Tin Research Institute is being used successfully in industry.

Enamelling—A large enamelling furnace is available, and special spraying rigs for applying vitreous enamels of high electrical resistance to complicated parts have been developed. In recent work in this field, pin-hole-free vitreous enamel coatings have been applied to copper, aluminium and stainless steel, using high frequency arcing as an acceptance test.

Chromium and Chromium-Base Alloys—Research is in progress on chromium metal and chromium-base alloys. Although the aim of the research is the development of alloys for very high temperature service, much more work on the pure metal itself remains to be done in order to understand and overcome its brittleness. This is achieved in various degrees by purifying—especially from nitrogen and carbon—and also by previous warm working treatments and heat treatments. Progress is being made with the extrusion of the pure metal and its alloys using



Apparatus for melting by electron bombardment

techniques similar to those which have been developed for molybdenum.

Ferrous Metallurgy—Research is in progress on low alloy and coated steels for high temperature service. During this work low alloy silicon aluminium steels with oxidation rates similar to that of 18/8 stainless steel in the 600-950° C. range have been developed. In recent work the effect of higher aluminium contents is being studied. Other work in the ferrous field has included a study of the effect of composition and structure of cast iron on its resistance to corrosion by sulphuric acid. A study has recently been made of the influence of production variables on the quality of cast iron shot.

Liquid Metals—Following X-ray diffraction work on relatively "simple" liquid metals like sodium and potassium, the breakdown in structure with temperature of "molecular" liquids such as gallium, bismuth and tin is being studied. In addition, interesting results are being obtained on alloys such as gold-tin, in which a high degree of order is detectable.

Ancillary Services

Although its primary function is to carry out research of a relatively long-term nature, the Institute undertakes analysis, testing, the investigation of service failures, consulting work, and short term investigations into industrial problems of a technical nature. The demand for this type of service is steadily increasing, and over 2,300 short-term investigations have been completed. The Institute is A.I.D. approved for mechanical testing, chemical analysis and weld examination, and also undertakes spectrographic analysis. Many of the short-term investigations are concerned with service failures in manufactured components such as gears, gear cutters, crankshafts, electrical switches and contacts, springs, bearings and bushes, but an appreciable number of problems of a non-metallurgical nature have also been dealt with.

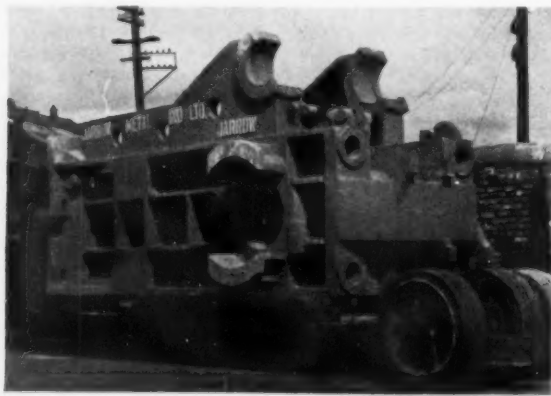
Socio-Industrial Venture Comes of Age

The Renaissance of Jarrow

A QUARTER of a century ago the name Jarrow was symbolic—symbolic, unfortunately, of the human misery resulting from the industrial depression. Conditions were not good in the country as a whole, but Tyneside in general and Jarrow in particular were especially hard hit. The one great industry in Jarrow, created by Charles Mark Palmer in the middle of the last century—that of shipbuilding—had closed down, and some 80% of the working population were without jobs, and had been for a considerable time.

In 1934 the late Sir John Jarvis, Bt., at that time High Sheriff of Surrey, became seriously interested in the plight of the people of Jarrow, and founded the "Surrey Fund" which raised some £40,000 by voluntary contributions. Instead of the money being distributed as charitable gifts, it was used to give back to the workers their self-respect. Paint, wallpaper and other things were provided for them to use themselves to brighten up their homes and clubs, and the men of Jarrow were employed on a monthly basis on public works such as the Monkton Dene Park, and paid modest sums for their services. Money was also spent on dredging the deep water berth alongside the quay, so that it was possible at a later date to carry out large shipbreaking operations.

The success of these early schemes encouraged Sir John to re-establish industry in the town, and with assistance from the Treasury two now well-known organisations were set up—Jarrow Tube Works, Ltd., and Jarrow Metal Industries, Ltd. The former is now a wholly-owned subsidiary of Tube Investments, Ltd., and the latter is associated with Armstrong-Whitworth (Metal Industries), Ltd. The official opening of the two works took place on Friday May 27th, 1938, when Her Grace, The Duchess of Northumberland started the machinery at the Tube Works and Lady Jarvis performed the opening ceremony at the Metal Industries Works.



A 37-ton press frame leaving the works of Jarrow Metal Industries, Ltd.

Originally designed for the production of tubes from 2½ in. to 3½ in. O.D. by the rotary piercer and pilger processes, the Tube Works have since been adapted to roll sizes as small as 2 in. and up to a maximum of 4½ in. O.D., with thicknesses from 10 s.w.g. to 1 in. or more. Since 1938, over 230,000 tons of tubes have been made, and in addition to this tonnage of hot finished tubes, 33.5 million feet of cold drawn tube have been produced. An appreciable proportion of this output has been in heat resisting alloys, which are now one of Jarrow's specialities.

Since the first cast of steel was made in March 1938 in the 15 ton Tagliaferri 3-phase electric arc furnace, Jarrow Metal Industries have made over 400,000 tons of steel for their various products, which include rolls, steel castings and ingots. Expansion and improved facilities have been introduced throughout the twenty-one years, and the firm is to-day capable of producing steel to most British Standard specifications. These include carbon steels, creep-resisting steels, pearlitic and austenitic manganese steels, chromium and stainless steels, and heat-resisting steels. Cast steel rolls of all types are produced up to 45 tons finished dressed weight for the steel and non-ferrous industries, and the castings include propellers, anchors, turbine, and valve castings, and frames, cylinders, crossheads, rams, etc., for large press construction; ingots are supplied up to 15 tons in weight.

To-day, Jarrow Metal Industries is equipped with three basic electric arc furnaces with a combined melting capacity of 55 tons. The foundry is equipped with modern sand mills, sand slingers and moulding machines, and heat treatment is carried out in a battery of pyrometrically controlled furnaces capable of accommodating material up to 25 ft. in length × 8 ft. 6 in. wide × 6 ft. high. In the dressing shop, a Hydroblast plant, a metallic shot blast plant, and a two-table Wheelabrator are available for cleaning castings. Other fettling equipment includes modern chipping hammers, grinders, Linde burners and air/carbon arc burners.

The research and inspection departments are particularly well equipped. The laboratories, which have recently undergone extensive improvements, are equipped for rapid and accurate chemical analysis, mechanical testing, the determination of the magnetic properties of materials, and metallographic work. A range of non-destructive testing techniques is used by the inspection department, the equipment including a Cobalt 60 isotope source for gamma-radiography, and electromagnetic and ultrasonic flaw-detecting apparatus.

To-day, Jarrow will bear comparison with any other town of its size in the country for progress and prosperity. This happy state of affairs is due in no small measure to the way in which these two concerns—which have given employment to more than 800 people over the years—showed to the world what Jarrow could do, and thereby encouraged many other companies to establish works in the area.

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NEWS AND ANNOUNCEMENTS

Electron Microscopy Symposium

A ONE-DAY Symposium on "The Application of Thin-Film Techniques to the Electron-Microscopic Examination of Metals," arranged by the Metal Physics Committee of The Institute of Metals, will be held on Thursday, 12th November, 1959, at the Royal Institution, 21 Albemarle Street, London, W.1, beginning at 9.30 a.m. Visitors will be welcome and tickets are not required. The following eight papers will be presented and discussed:

"Techniques for the Direct Examination of Metals by Transmission in the Electron Microscope," by P. M. KELLY and J. NUTTING (University of Cambridge).

"An Outline of the Theory of Diffraction Contrast Observed at Dislocations and Other Defects in Thin Crystals Examined by Transmission Electron Microscopy," by M. J. WHELAN (Cavendish Laboratory, Cambridge).

"Observations of Dislocations in Metals by Transmission Electron Microscopy," by P. B. HIRSCH (Cavendish Laboratory, Cambridge).

"The Observation of Anti-Phase Boundaries during the Transition from CuAu I to CuAu II," by D. W. PASHLEY and A. E. B. PRESLAND (Tube Investments Research Laboratories, Cambridge).

"Electron-Microscopic Studies of Precipitation in Aluminium Alloys," by R. B. NICHOLSON, G. THOMAS, and J. NUTTING (University of Cambridge).

"Electron-Microscopic Observations on the Recrystallization of Nickel," by W. BOLLMANN (Battelle Memorial Institute, Geneva).

"The Martensite Transformation in Thin Foils of Iron Alloys," by W. PITSCH (Max-Planck-Institut für Eisenforschung, Düsseldorf).

"The Growth, Structure and Mechanical Properties of Evaporated Metal Films," by G. A. BASSETT and D. W. PASHLEY (Tube Investments Research Laboratories, Cambridge).

Copies of the papers, published in the August issue of the *Journal of the Institute of Metals*, are obtainable (price £1 post free, to non-members), from the Secretary, The Institute of Metals, 17 Belgrave Square, London, S.W.1. In connection with the meeting, an informal conversation will be held at 17 Belgrave Square, London, S.W.1, on the evening of Wednesday, 11th November. Tickets (price 6s.) can be obtained from the Secretary.

Iron and Steel Prices

THE Iron and Steel Board have recently announced changes of detail in some of their maximum price schedules, which will, however, have no appreciable effect on the overall level of iron and steel prices. Certain weight and quality extras chargeable by plate producers have been reduced, and minor changes have been made in alloy forging ingot extras applicable to special purpose steels. The extras chargeable for small quantity sales by producers trading as stockholders have been increased in some cases. Alterations have been made in the basic hematite and foundry iron schedules, and in particular prices will in future include delivery from railway stations to works without additional cost to consumer. The formula under which the prices of strip mill tinplate are varied in accordance with the average price of tin has been modified so as to eliminate narrow changes in the prices of tinplate. In consequence the prices of strip mill tinplate

which would have risen on October 1st under the previous formula have not done so. Maximum price control has been removed from the relatively small quantities of tinplate, tinned sheets and blackplate still produced in hand mills. The alterations became effective from September 28th.

Cast Iron Lecture Course

THE British Cast Iron Research Association, jointly with the Metallurgy Department of the Royal College of Science and Technology, Glasgow, has organised a series of lectures to be given in Glasgow at the Royal College during October to December, 1959. This course of lectures, under the title "Cast Iron as an Engineering Material" is intended primarily for the engineer and designer: it aims to instruct or re-inform them on the metallurgy and properties of the material, and to indicate correct principles of design from the aspect of both the foundryman and the user of the casting.

Cast iron is one of the oldest and one of the most versatile of the cast materials. Research and industrial experience during the last twenty years have led to closer control in production methods, and the performance of a correctly specified and designed casting for a given service can be predicted with considerable accuracy. The improvement in the engineering status of cast iron is reflected in the more exacting requirements of British, continental and American standards, and in the range of new materials now available to the engineer.

The programme comprises fourteen lectures, two to be given on each successive Thursday afternoon from October 22nd to December 3rd, 1959. With one exception, the lectures will be undertaken by B.C.I.R.A. staff. Further particulars and enrolment forms can be obtained from the Secretary, The Royal College of Science and Technology, Glasgow, C.1 (tel: Bell 4400). The enrolment fee is £3 3s. for the course of fourteen lectures.

Induction Heating Course

A COURSE for users and potential users of induction heating is being organised by the R. F. Heating Division of Pye, Ltd., and will commence at 2 p.m. on Tuesday, December 1st, 1959 and conclude at 6 p.m. on Wednesday December 2nd. It will cover the theory of induction heating, the operation of the generator, and the design of coils for hardening, annealing, tempering, brazing, soft soldering and special applications. The course itself is free, but members attending will be required to pay for their living accommodation. Further details of the course, requests to attend, etc., should be addressed to Mrs. E. Raeburn, Pye Process Heating, 28, James Street, Cambridge.

Corrosion in the Petroleum Industry

A SYMPOSIUM on "Corrosion Problems of the Petroleum Industry", organised jointly by the Institute of Petroleum and the Corrosion and Chemical Engineering Groups of the Society of Chemical Industry, will be held on Thursday and Friday, November 26th and 27th, 1959, in the Grand Council Chamber, Federation of British



Demonstration of Metrovick submerged arc automatic welding machine at an exhibition organised by the Heating and Welding Department of the A.E.I. Transformer Division, and held in Newcastle upon Tyne at the beginning of last month. It is to be repeated at the Midlands Electricity Board Showrooms, Aston, Birmingham, from October 19th to 30th, 1959.

Industries, 21 Tothill Street, London, S.W.1. A reception for participants in the symposium and their guests will be held at the Washington Hotel, Curzon Street, W.1 on the evening of Wednesday, November 25th. Members of the Society of Chemical Industry or the Institute of Petroleum will pay a fee of £1, other participants £3. Further particulars and registration forms may be obtained from the Assistant Secretary, Society of Chemical Industry, 14 Belgrave Square, London, S.W.1.

High Temperatures by Electricity

THE Morgan Crucible Co., Ltd., is co-operating with the North Western Electricity Board in practical demonstrations of high temperature equipment at the Board's showrooms in the Town Hall Extension, Manchester, from October 26th to 30th, inclusive. Each day the exhibition will be open from 9.30 a.m. to 5.30 p.m., and specialist staff will be in attendance to discuss visitors' problems. A number of high temperature furnaces illustrating the versatility of Crusilite furnace heating elements will be operating at temperature throughout the exhibition, and the complete range of elements now available, associated refractory materials, and experimental mock-up furnaces, will also be displayed.

Junior Engineers in Scotland

A MEETING of Junior Engineers, organised by the Iron and Steel Institute, will be held in Glasgow on Wednesday and Thursday, October 28th and 29th, 1959. The object of the Meeting is to provide an opportunity for the younger engineers and operators in the iron and steel and associated industries to discuss engineering problems, and to visit works of interest. The latter will include the General Ore Terminus, Clyde Iron Works, Clydebridge Steelworks and Ravenscraig Iron and Steelworks of Colvilles, Ltd.; the Clydesdale Steelworks and Tube Works of Stewarts and Lloyds, Ltd., and the works of the North British Locomotive Co., Ltd.

Personal News

MR. S. R. HOWES, O.B.E., retired from the board of The United Steel Cos., Ltd., at the end of last month. He was appointed a Director in June, 1956, and assumed special responsibility for the Company's then newly-created Department of Operational Research and Cybernetics. Mr. Howes was formerly a Director and General Manager of Samuel Fox and Co., Ltd., a United Steel subsidiary: he remains Managing Director of Templeborough Rolling Mills, Ltd., in which the Company has a financial interest.

MR. R. S. MEDLOCK, Technical and Home Sales Director of George Kent, Ltd., has been elected President of the Society of Instrument Technology.

THE Development Branch of the Development and Research Department of The Mond Nickel Co., Ltd., has recently been reorganised into four divisions under the General Managership of Mr. F. DICKINSON, a Director of the Company. The divisions and their managers are: Ferrous—MR. W. W. BRAIDWOOD, Non-Ferrous—MR. J. HINDE, Applicational Engineering—DR. A. B. EVEREST, and General (including Plating, Chemical Products and Nuclear Power)—DR. E. C. RHODES. MR. L. W. JOHNSON, at present Assistant Manager of the Department, relinquished his appointment at the end of September, having reached retirement age.

MR. A. MOORE, "Kareth," 20 Eden Crescent, Leeds, 4, has been appointed Area Technical Sales Representative for Brayshaw Furnaces, Ltd., in the territories comprising Northumberland, Durham, Yorkshire, Leicestershire, Derbyshire, Northants and Lincolnshire.

MR. H. G. STERN, for the past six years Head of the Control Department has been appointed a Director of Londex, Ltd.

MR. P. B. FORREST has been appointed Works Manager of Fawcett Preston and Co., Ltd., the Metal Industries' engineering subsidiary.

MR. J. E. ROBSON, M.C., of Samuel Osborn and Co., Ltd., Sheffield, has been appointed to the Board of the Company.

WALKER, CROSWELLER AND CO., LTD., manufacturers of Arkon industrial instruments, announce that their Representative in the Midlands, Mr. W. W. NICHOLAS, has left them to take up a teaching appointment at Birmingham Technical College. He will be succeeded at Walker Crossweller by MR. E. W. TINSLEY, who comes to them from Radiation, Ltd., where he was concerned with appliance development.

Obituary

WE regret to record the death on September 5th of MR. W. J. WOOD, Chief Assistant for the organisation of the Metropolitan-Vickers Works School, with responsibility for recruitment and selection of craft apprentices. Joining the Company as an apprentice instructor in 1920, he was associated with the pioneer developments of industrial education under Sir Arthur Fleming, former Director of Research and Education, and the late Mr. K. R. EVANS. With almost forty years of work in the Education Department he will be affectionately remembered as "Tiny" Wood by thousands of M-V trained engineers in almost every part of the world.

RECENT DEVELOPMENTS

MATERIALS : PROCESSES : EQUIPMENT

Mineral Insulated Thermocouples

BRITISH INSULATED CALLENDER'S CABLES, LTD., who have for some time been manufacturing mineral insulated cables are now applying the principle of mineral insulation to thermocouple production. The thermocouples consist of two dissimilar wires, welded together at the hot junction, embedded in highly compressed magnesium oxide insulant, and completely encased in a seamless, circular, stainless steel sheath. The positive conductor is of nickel-chromium alloy and the negative conductor of nickel-aluminium alloy.

The thermocouples are produced in standard overall diameter sizes of 0.122 in. and 0.060 in., but intermediate sizes are also available. In the standard hot junction, the conductors are welded together, and the sheath is welded over to form a hermetic seal completely insulated from the conductors. This does not increase the overall diameter of the cable nor make installation any more difficult. For the first time, thermocouples of all sizes down to 0.060 in. diameter can be supplied with an insulated junction. At the cold end, a seal is fitted to prevent the insulant from absorbing moisture. The seal body is externally threaded to facilitate plain hole entry into junction boxes and instrument casings, where it can be held in place by lock-nuts. The cold end assembly will operate at temperatures up to 150° C. Alternative arrangements at both hot and cold ends can be made to suit customers' requirements.

These thermocouples are available in lengths up to 100 yds., and the excellent conductivity arising from the small volume and high density of insulant gives rise to rapid thermal response. A recent addition to the range has been made possible by drawing on established cable manufacturing techniques. By laying up mineral insulated stainless steel sheathed thermocouples with stainless steel strands, a multiple flexible thermocouple of small overall diameter and high tensile strength is produced.

British Insulated Callender's Cables, Ltd., 21 Bloomsbury Street, London, W.C.1.

Nickel-Chromium Casting Alloy

NIMOCAST 713C nickel-chromium casting alloy has been added to the range of Nimocast alloys now available in the U.K. It is the Wiggin-made equivalent of Inconel 713C, an investment casting alloy for high temperature service developed in the research laboratories of The International Nickel Company, Inc., and already specified for the turbine rotor blading of a number of U.S. gas turbine aero-engines.

Nimocast 713C is one of a number of complex high nickel casting alloys developed in recent years for high temperature turbine blading. It combines outstanding strength with excellent resistance to thermal fatigue at temperatures up to 980° C., and is therefore of interest as a possible alternative to wrought alloys, which offer difficult production problems when developed for service at such elevated temperatures. In the U.S.A., where cast rotor blades are considered acceptable for production

TYPICAL PROPERTIES OF NIMOCAST 713C

Material	Testing Temperature (° C.)	Stress to Rupture (tons/sq. in.) for a life of			
		30 hr.	100 hr.	300 hr.	1,000 hr.
As Investment	815	28.0	24.5	21.5	18.0
	870	19.5	16.5	14.5	11.5
	940	(12.5)	(10.5)	(8.5)	—
	980	8.5	7.2	—	—
As Investment Cast in Vacuum	870	22.8	19.0	16.5	14.5
	940	14.0	12.0	10.0	8.5
	980	10.5	8.7	7.5	6.8

engines, and are in fact used in substantial numbers, this alloy is now well-established and has won a reputation for reliability under arduous service conditions.

Nimocast 713C is readily castable and develops its best properties when cast in vacuum. Alternatively, it can be cast under a protective atmosphere such as argon. A heat treatment of two hours at 1,170° C. followed by air cooling is beneficial, but is not essential unless the maximum mechanical properties are required. Typical properties of the as-cast material are given in the table.

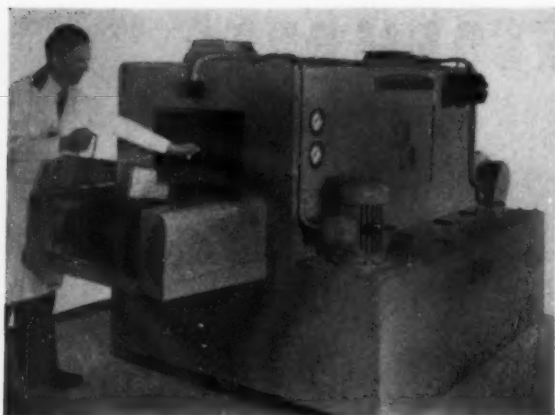
Henry Wiggin & Co., Ltd., Thames House, Millbank, London, S.W.1.

Metal Cleaning Equipment

A COMPLETELY new range of standard industrial spray-washing equipment has just been introduced by the Electro-Chemical Engineering Co., Ltd., comprising single and two-stage machines fitted with either mesh belt, flight bar or overhead monorail conveyors. All machines can be supplied with drying sections, if required, and they incorporate several special features not previously available in standard industrial cleaning equipment. Particular attention has been given to those design aspects affecting accessibility, maintenance and safety.

In operation, the parts to be cleaned are loaded on to the conveyor and carried through a steel canopy where they are sprayed from top, sides and bottom with pump-circulated cleaning agent from stainless steel nozzles adjusted to give the maximum coverage of the work. Full width, roller mounted sliding doors and a translucent glass fibre roof hatch improve access to the canopy interior. Vertical monoblock pumps are employed, which eliminate external pipework and gland spillage. The two canopy openings are provided with internal lip extraction ducts, connecting into fume extraction stubs mounted on top of the machine. Emergency stop buttons are fitted to each end of the conveyor, the drive mechanism of which is fully guarded.

The solution tank is fitted with removable covers and a quick-release door, with a space between the tank bottom and the shop floor to give access for hosing down. A quick-filling water service connection is provided, with a float valve to maintain the cleaning agent at constant level during operation. An overflow weir prevents overfilling, and enables floating contaminants to be removed from the surface of the cleaning agent: a sloping bottom and drain sump allow complete emptying of the tank.



Single-stage flight-bar conveyor industrial spray washing machine.

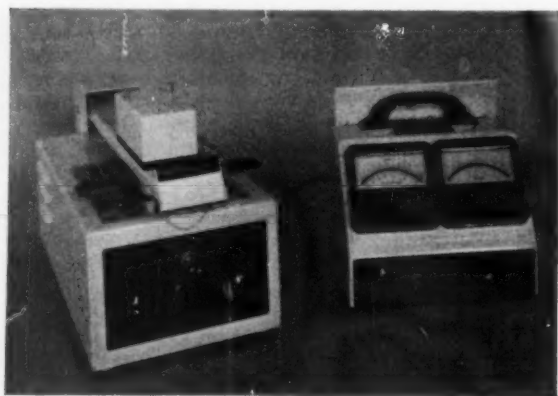
Pump output is rated to permit solids to settle before recirculation of the cleaning agent, and delivery is controlled by a hand valve: the suction side is protected by detachable strainers. The pressure and temperature gauges are flush mounted in a sealed chamber, which protects them from physical damage and ingress of water vapour. All exposed metal parts of the machine are phosphated and painted with two coats of alkali-resistant enamel. Heating is by gas, high pressure hot water, steam or electricity: thermal insulation is available as an optional extra.

The standard range of sizes includes conveyor widths of 18 in., 24 in. and 30 in. for the belt and flight bar machines. Variable speed drives and special heavy duty conveyors are available as optional extras.

Electro-Chemical Engineering Co., Ltd., Sheerwater, Woking, Surrey.

Personnel Protection Densitometer

This instrument has been designed for the rapid and accurate measurement of density of processed protection film badges used by radiographers. It enables low levels of density to be measured with a high degree of accuracy and, furthermore, may be used as a precision general



Personnel protection densitometer for radiographic film badges.

purpose densitometer. The instrument comprises two units, a controlled light source and a vacuum photo-electric cell with a D.C. amplifier.

The light unit is fed from a constant voltage transformer and has a coarse and a fine rheostat fitted for the control of level of illumination. The measuring table is of sufficient area to enable large X-ray plates to be measured also. The standard instrument is fitted with two viewing apertures, one $\frac{3}{16}$ in. in diameter and the other $\frac{1}{16}$ in. in diameter. With the large aperture, densities up to 6 can be read easily.

The output from the photocell is fed into a highly stable D.C. amplifier fitted with two inter-calibrated meters, one scaled to read over the density range zero to 2.0 and the other scaled to read zero to 0.5. A switch is provided to enable the user to change from one meter to the other. The D.C. amplifier is fitted with a range switch, which extends the scale of the standard meter by a factor of 3, thus leading to higher reading accuracy on high density. The instrument is portable and easy to use, which renders it suitable for semi-skilled operators.

Baldwin Instrument Co., Ltd., Brooklands Works, Dartford, Kent.

Lead Bricks

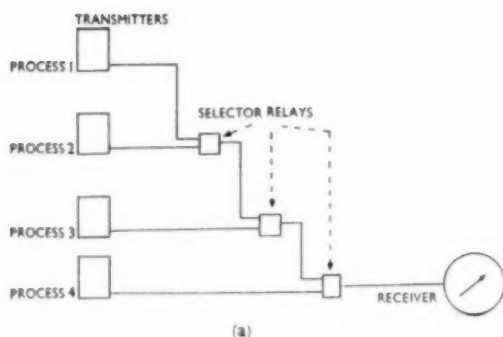
A new lead brick for use in shielding personnel against alpha, beta and gamma rays has been produced by a special pressure moulding technique by British Lead Mills, Ltd., a member of the Firth Cleveland Group. The company was approached by the U.K.A.E.A. Harwell some time ago and after a period of close liaison with Harwell a pressure moulded lead brick was produced by the company at Welwyn Garden City. This brick was accepted by Harwell as a standard for all future requirements. The company claims to be the first in the field with lead bricks produced by pressure moulding, which ensures the greatest density and freedom from porosity and inclusions within the fine limits permissible under Harwell's very rigid specification.

These bricks are produced in 2 in. and 4 in. thicknesses in the form of standard, corner, top and bottom bricks, and also half or even quarter bricks, which can be built up into a complete surround by virtue of their interlocking nature, to give the maximum thickness at any point of 2 in. or 4 in. or multiples thereof.

British Lead Mills, Ltd., Byron House, 7/9, St. James's Street, London, S.W.1.

Pneumatic Selector Relay

A RELAY which will accept two signals in the 0-100 lb./sq. in. range and always pass the higher signal has been added to the range of pneumatic relays produced by Sunvic Controls, Ltd. Known as the Model 58S, it is intended to complement the Sunvic Model 61F booster relay, which is a minimum auto-selector. These two relays combined have a variety of applications. For instance in a system measuring several different values of the same parameter, it may be necessary to know the highest or lowest value; the extreme pressure is always indicated on a receiver. These two relays may also be used for over-ride control in which one variable is controlled, the rest being tied in through the selector relays. If any of these other variables reaches an extreme value, its controller output is transmitted to the control valve:



this system eliminates the use of extra control valves. The Model 58S relay will operate in ambient temperature from -40°F. to $+180^{\circ}\text{F.}$, and the maximum operating pressure is 100 lb./sq. in. As there is no re-transmission, there is no time delay.

Sunvic Controls, Ltd., P.O. Box 1, Harlow, Essex.

Press Clutch Valves

COMPLETE fail-to-safe features for any electro-pneumatic system can be achieved with the I.G.E. patent Saffail valve, which is designed so that there is five times the force available to move the operating spool from "on" to "off" than is available to move it from "off" to "on." This differential is checked electrically every operation, and when the differential drops, through any fault, the valve is locked "off" until the fault is rectified. For the control of electro-pneumatic power press clutches, I.G.E. have designed around this valve a control system, which gives complete self-checking of all electrical circuits as well. Thus the complete system provides the best safeguard available against uncovenanted or repeat strokes of the press. The valve is manufactured to I.G.E. specification by Messrs. TAL Numatics, whose own special spool construction is incorporated.

J. P. Udal, Ltd., I.G.E. Automation Division, Court Road, Birmingham, 12.

Atomic-Absorption Spectroscopy Equipment

ATOMIC-ABSORPTION spectroscopy is the measurement of the depletion of a beam of light as it passes through a population of free atoms—usually a flame. The depletion is due to the resonance of the light with the free atoms and occurs at distinct frequencies characteristic of the absorbing elements. Since an absorption line is extremely narrow, its intensity cannot be measured accurately against the background of a continuous spectrum; therefore monochromatic light at the resonance frequency must be used.

Atomic-absorption spectroscopy is an accurate and sensitive method of analysing metal samples in solution, and is particularly suitable for analysing samples that are not amenable to other methods of spectrochemical analysis. In atomic-absorption spectroscopy, as in flame photometry, a solution of the sample is sprayed into a flame, but it is the absorption of light by free metal atoms in the flame, and not the emission, that is measured. This method has many advantages: its accuracy is not affected by the temperature of the flame; it determines elements not excited in a flame; it is very

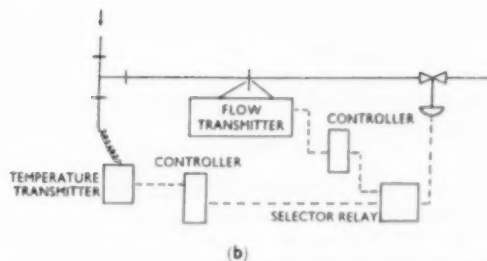


Diagram of pneumatic selector relay: (a) monitoring system; (b) override control.

sensitive; and it avoids the effects of inter-element interference.

The Hilger atomic-absorption equipment consists of a hollow-cathode lamp emitting light of the resonance frequency, and a specially designed flame-vaporiser. It is designed for use with the Hilger Uvispek spectrophotometer, but may be used with similar spectrophotometers. It is best used with recent models of the Uvispek, for then the spectrophotometer needs no modification, and may be used for normal spectrophotometry without dismantling the equipment.

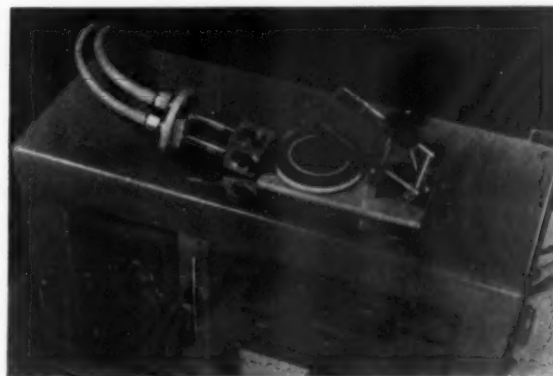
Hilger & Watts, Ltd., 98 St. Pancras Way, Camden Road, London, N.W.1.

Sigrist Photometers

SOUTHERN INSTRUMENTS, LTD. can now supply in the U.K. the Swiss Sigrist photometers, which may be divided into two main categories, for industrial and laboratory use, respectively. All types employ a design feature which enables continuous measurements of turbidity and colour to be carried out, independent of common sources of error such as variations in photocell sensitivity, light source intensity and amplifier gain.

One type of Sigrist industrial photometer uses flow cells for turbidity and/or colour measurements having a wide variety of finishes for corrosive liquids and gases. Other types enable turbidity measurements to be made from the surface of a liquid and colour measurements from a free flowing jet. Weather-proof cases make operation possible under the most arduous conditions.

The Sigrist laboratory photometer makes turbidity and colour measurements possible from static measuring vessels, and, due to its independence of instrumental



The Sigrist photometer.

variations, many samples may be continuously compared with a standard over long periods, without adjustments. The laboratory photometer may be fitted with a built-in monochromator and recorder if desired.

Southern Instruments, Ltd., Frimley Road, Camberley, Surrey.

Cupola Dust Collector

THE solids discharged to atmosphere with the stack gases may amount to as much as 10 lb./min. in the case of large cupola installations. It is not surprising, therefore that such emissions, which constitute a serious nuisance in the vicinity of the plant, infringe the requirements of the Clean Air Act. To meet the need for dealing with this problem, the Holmes-Schneible S.W. cupola dust collector has been introduced.

The S.W. collector consists of an outer shell and collection trough supported on an angle ring welded to the cupola stack. A deflector cone is supported from the collector shell in such a way that the cupola gases are confined to the annular passage between the edge of the cone and the collector shell. A non-clogging, adjustable distribution head of patented design distributes the water equally over the surface of the cone without the use of sprays. The gases thus pass upwards through a water curtain which removes the ash and cupola grit, the water and collected solids impinging on the inside of the collector casing and running down into a collection trough, from whence they drain to a Schneible recirculating and dewatering tank. An adjustable entrainment baffle is fitted in the collector to arrest droplets of water that would otherwise be carried away by the gas. The dewatered sludge is discharged automatically and the clarified water recirculated.

W. C. Holmes & Co., Ltd., Gas Cleaning Division, P.O. Box B7, Turnbridge, Huddersfield, Yorks.

Aluminium Bale-out Furnace

THE need for minimising heat losses in melting and holding at temperature aluminium for die casting prompted the design by Industrial Furnaces, Ltd., of a unit for this purpose. The illustration shows a furnace of the open-top type, but other models available include one fitted with a counterbalanced swing-clear lid.



Open top aluminium holding furnace

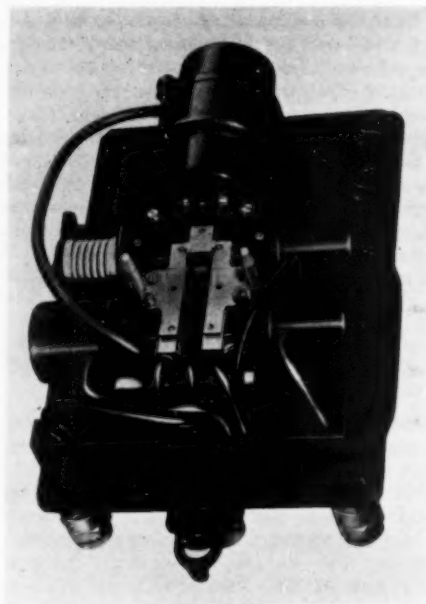
The furnace is electrically heated, and the special elements, supported in new type carriers, are designed to give a rapid and even heat input for melting and a low input for holding. The input is three-phase, and the heating rates can be varied to suit individual requirements: a typical unit will heat 300 lb. of aluminium from cold to 800° C. in 2½ hours. Temperature control is automatic, and rapid melting of aluminium make-up minimises loss of production time, and ensures a steady aluminium pouring temperature throughout the day.

Industrial Furnaces, Ltd., Kingswinford, Staffs.

Compensated Flow Measurement

THE size of an orifice plate for measurement of gas flow is calculated for one temperature and one pressure; it follows that any variation in either will affect the accuracy of the final reading. In most cases, variations are small and the effect on metering accuracy is negligible. However, instances do arise in which large changes in either pressure or temperature, or both, are encountered, and it is then necessary to apply a correction.

Honeywell Controls, Ltd., are now able to supply a system providing automatic compensation for either temperature or pressure, or both—using the Sorteberg Force Bridge pneumatic relay. This is essentially a multiplying and dividing unit which corrects the flow signal for pressure and temperature changes. The



The Sorteberg force bridge

pressure and temperature transmitters are calibrated from absolute zero to the maximum values for which compensation is required, expressed in absolute units. Transmission is such that zero absolute is 3 lb./sq. in., whilst the maximum reading gives 15 lb./sq. in. The flow transmitter is a standard differential converter, or similar transmitter, which does not incorporate square root extraction.

Honeywell Controls, Ltd., Ruislip Road East, Greenford, Middlesex.

CURRENT LITERATURE

Book Notices

FIFTH INTERNATIONAL CONFERENCE ON HOT DIP GALVANIZING

Edited Proceedings of the Conference held in Holland and Belgium in June, 1958. 354 pp., numerous diagrams and illustrations. Published by the Zinc Development Association, 34 Berkeley Square, London, W.1. 63s.

THREE hundred and fifty delegates from twenty countries attended the Benelux Conference in Hot Dip Galvanizing in 1958, when twenty papers by nineteen authors from eight countries were presented for discussion. These papers are printed as ten chapters in this book, and each chapter concludes with an edited account of the discussion which followed. In a number of cases, important written contributions received since the Conference have been included to bring the book completely up to date. Many aspects of interest to both specialist and general galvanizers are described, and users of galvanized products will find the chapters on painting and thickness testing of considerable interest.

Chapter 1 consists of three papers on pickling and fluxing. In the first, "Efficiency of Hydrochloric Acid Pickling," F. Sjoukes (Netherlands) discusses the factors which influence acid loss and pickling time: formulae and test methods are given in appendices. R. Haarmann (Germany) describes the Ruthner process in his paper "Reclamation of Hydrochloric Acid from Spent Pickle Liquors," and performance data and operating costs are given for two plants. It is estimated that the process is economic for throughputs of a thousand tons of steel per month, but future developments will probably enable smaller installations to be used profitably. J. Hille and W. Durrwachter (Germany) present a paper entitled "The Behaviour of Molten Fluxes for the Hot-Dipping Process in Zinc, Tin and Lead." It describes experimental work which seeks to break away from the merely qualitative assessment of flux behaviour.

Modern methods of heating are rapidly replacing the old coke-fired installations throughout the galvanizing industry, and very wide interest is shown in the new developments. The four papers on bath heating are collected in Chapter 2. J. G. C. Pope (U.K.) presents a comprehensive account of oil firing, in which he discusses the properties of fuel oils, setting designs, temperature control and control systems, oil storage, oil heating, filters, burners, combustion chambers, stack losses, etc. R. Haarmann describes a top heating system for a wire galvanizing plant, discussing alternative hood positions, temperature distribution and ash formation, and comparing them with side fired baths. In his paper "The Line Frequency Induction Furnace for General Galvanizing," H. Chapman (Canada) outlines the induction heating of a galvanizing bath used by his company for hand dipping, and A. Parent (Belgium) describes an electric resistance heating system, in which the elements are immersed in molten lead surrounding the bath.

P. Hoesli and E. C. Mantle (U.K.) describe their researches on zinc efficiency in the paper "Survey of Galvanizing Processes with Particular Reference to the Efficiency of the Use of Zinc," which forms Chapter 3. Four kinds of galvanizing process—the dry process, the wet process, the old dry process and the modified

wet process—are defined and their efficiency assessed in terms of zinc consumption; zinc losses in ash, flux skimmings and dross; coating weight; flux consumption and labour.

Chapter 4 consists of H. Bablik's "Comparison Between the Cook-Norteman and Sendzimir Process." This paper, and those on wire galvanizing, were included to cater for the interests of specialist galvanizers attending the conference. Dr. Bablik surveys the history of strip galvanizing and deals in an elaborate way with the mechanical properties of the product, as well as the thickness, uniformity and composition of the coating. The factors affecting these properties are considered with characteristic thoroughness.

"The Effect of Painting on the Corrosion Resistance of Galvanized Steel" by P. Morisset (France) forms the subject matter of Chapter 5. An attempt is made to evaluate the performance of painted and unpainted galvanized samples by using accelerated corrosion tests. Colour photographs of panels subjected to the salt spray test and the Industrial Building Cycle are included, and the article concludes with a discussion of the nature of the additional protection given to galvanized steel by paint.

Chapter 6 consists of two papers on wire galvanizing. B. Ulrich (Netherlands) on the one hand, and the Italian team of F. Baldi, G. Garbelli and A. Pirozzi on the other discuss the effects of hot dip galvanizing on the mechanical properties of steel wire.

Three papers on the influence of bath and steel composition on galvanized coatings make up the seventh chapter. J. J. Sebesty and J. O. Edwards (Canada) discuss the influence of aluminium, lead and iron; J. J. Sebesty studies surface carbides, differential steel attack and pore formation in the galvanizing process, and W. Gerber, R. Gloor and H. Oertli (Switzerland) consider the influence of steel quality, particularly the silicon content, on coating thickness.

The importance of coating thickness has led to extensive efforts to develop a non-destructive method of measurement. In Chapter 8, after describing various destructive methods of measurement, W. Peppler (Germany) goes on to discuss, at length, magnetic and electro-magnetic methods, and to consider the influence of the magnetic permeability of the basis steel, and of the area, thickness and curvature of the sample. He concludes that there is no insurmountable objection to the use of magnetic gauges on heterogeneous coatings such as those obtained by hot dip galvanizing.

The drive for greater efficiency in industry since the war has led to increased attention to materials handling, the subject matter of Chapter 9. A. G. Northcott of the Zinc Development Association has spent many years applying work study techniques to the galvanizing industry, and sets forth his conclusions in his paper "Materials Handling In Galvanizing." A subsidiary paper by C. van Kempen (Holland) approaches the problems practically and discusses handling in relation to costs.

The final chapter of the book consists of two papers on the theme of the Brussels World Fair, under whose auspices the conference was held, "Human Progress Through Technical Progress." R. Palmers (Belgium)

describes the improvements in working conditions in the galvanizing shop which have resulted from technical progress, and R. L. Stubbs, Director of the Zinc Development Association, shows, in his historical survey, the way in which developments in galvanizing have contributed step by step to human progress throughout the last 150 years.

Trade Publications

THE properties and fabrication of high-strength low-alloy steels are receiving considerable attention in connection with the increasing use which is being made of the materials in aeronautical applications. Of interest in this respect are nine papers presented, in 1958, at an American symposium on High-Strength Steels for Aircraft, abstracts of which form a feature of the July issue of *The Nickel Bulletin*. The other items of the issue are relevant to many of the industrial fields in which nickel-containing materials find application. Reference is made, for example, to papers on extraction and determination of nickel and on direct-rolling of nickel strip, while abstracts on nickel plating cover investigations concerned with the influence of plating variables and the quality of the deposits. Structural studies on nickel-containing non-ferrous alloys are noted, and in the section on nickel-iron alloys attention is directed to papers presented at a conference on magnetic materials. Cast-iron interests are represented by items on the thermal and elastic properties of various grades of iron, the use of Ni-Hard for mill liners, and the applications of S.G. iron in steelworks. The largest section of the issue, that concerned with nickel-containing heat- and corrosion-resisting materials, contains forty abstracts covering papers on properties, fabrication and applications. The issue also includes the quarterly review of recent patent literature.

WE have recently received from The Incandescent Heat Co., Ltd. three brochures dealing with foundry plant. Two of them are concerned with equipment manufactured under licence from the Whiting Corporation, Harvey, Illinois, U.S.A. Publication F.P.1 is concerned with cupolas and chargers and is extensively illustrated with views of recent installations with melting rates ranging from 4 to 20 tons per hour. Where a low melting rate is required, the Whiting Cupolette is available, and this too is referred to in the brochure. Incandescent-Whiting wet dust arresters are featured in publication F.P.2, together with associated sludge handling plant. Considerable savings are claimed for the use of hot blast in cupola operation, and Incandescent brochure F.P.3 describes air heating equipment in which fuel is completely burnt in a high intensity combustion chamber, mixed with a proportion of waste gases and passed through a heat exchanger. The blast air to be heated makes a single pass through a bundle of straight tubes in the heat exchanger.

ARALDITE epoxy resins today enjoy wide acceptance and a reputation for remarkable and consistent performance, and designers in many spheres of engineering—aircraft, marine, automobile and electrical—regularly specify them. Almost every material in common use can be bonded, and these resins are being used ever more widely throughout industry and in the domestic field.

A number of different types is available and Ciba (A.R.L.) Ltd., Duxford, Cambridge, have now published a booklet which will be of value for those wishing to select the best adhesive for use with particular materials and for special applications. Recommendations are also made for the pretreatment of materials, where necessary.

FEATURED in the June, 1959 issue of *The English Electric Journal* is an article on non-destructive testing in electrical engineering, by D. J. Griffiths, Works Radiologist at Stafford. In addition to radiographic methods, the author discusses ultrasonic flaw detection, magnetic crack detection, magnetic sorting methods, conductivity measurements, thickness measurement, leak detection and, more briefly, techniques such as sulphur printing, the dye penetrant process, electrostatic crack detection, and the brittle lacquer method of stress measurement, and concludes by referring to the possibilities of new developments such as the image intensifier and xeroradiography.

To the wide range of technical literature issued by the Copper Development Association, there has now been added a publication covering the comparatively new, but extremely interesting, subject of the production of brass and copper-base alloy components by hot pressing and forging. The advantages to be gained by the use of these methods when applied to brass and other copper-base alloys are fully described, together with the various stages of manufacture. Details are given of the relevant material properties, and of the presses and tools employed; some useful hints on design are also included. With the co-operation of several specialist organisations, it has been possible to provide a considerable amount of data which has never before been dealt with so comprehensively under one cover.

THE increasingly severe conditions of modern blast furnace operation call for new and better refractories. To meet this need, General Refractories, Ltd., have made improvements in the existing Foster range, and introduced completely new types of blast furnace refractories. A new leaflet issued by the company lists the available grades—which include Numax, Sillmax 1, Foster High Grade S, Foster Crown S, Foster Carbon, and Amberlite Insulating—and shows by coloured sketches the locations in which each grade is used in both the blast furnace and the hot blast stove.

THE principal article in the June issue of *The Wilde-Barfield Heat Treatment Journal* is a resumé of a lecture on modern heat treatment equipment given by Mr. L. G. W. Palethorpe, the company's chief metallurgist, during a course on modern heat treatment practice organised by the Department of Mechanical Engineering at the College of Technology, Bristol. Other features deal with mains frequency crucible type induction melting furnaces; an enamelling furnace at West Bromwich; and a sealed quench bright hardening unit at Wolverhampton.

DANGER attends the use of steel tools in an area of potentially explosive material, and special non-sparking tools have been developed for service in such environments. A brochure recently issued by Charles Carr, Ltd., Grove Lane, Smethwick 40, Birmingham, details the Lily brand aluminium bronze made by the company and available as shovels, scrapers, crow bars, picks, caulking tools, hammers, chain, spanners, chisels, wedges, screw-drivers, pliers, clamps and spikes.



the one that got away...

Friday fish, to find one of the few anglers whose hooks are not hardened in Birlec furnaces! For all small steel parts needing accurate production hardening and tempering, Birlec shaker hearth furnaces are pre-eminently suitable.

BIRLEC SHAKER HEARTH furnaces are available in standardised capacities with appropriate non-scaling, non-decarburising atmosphere control.



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Lighting-1

Good lighting affects productivity in three ways, (1) by directly increasing the speed of working and reducing errors and wastage, (2) by improving overall factory efficiency through better supervision and housekeeping, (3) by providing better working conditions, thus improving labour relations and avoiding frequent changes of workpeople.

It is not possible to judge by the eye alone whether the lighting in any factory is good enough to serve these purposes because the eye has a tremendous range of adaptation (vision of some kind is possible within an illumination range of 1,000,000 to 1) and is hence an unreliable measuring instrument. Severe mental and eye strain or unconscious slackening of working speed may occur under lighting which *appears* to be adequate.

The only reliable way to appraise factory lighting is to conduct a lighting survey using a lightmeter, an inexpensive instrument which measures the actual illumination available. These figures can then be compared with official recommended values for the various tasks.

Lighting Survey

A methodically conducted lighting survey is the first step towards achieving good productive lighting. The average artificial illumination at working height should first be measured. This requires a number of readings at various positions relative to the lighting fittings, particularly beneath and between fittings and by the wall. The average of the readings should then be compared with the illumination values recommended by the Illuminating Engineering Society. The following is a general guide to illumination requirements.



NATURE OF WORK, PROCESS OR MATERIAL	ILLUMINATION LUMENS/SQ. FT.
Rough or routine work. Large detail. Medium to light material of good contrast.	7
As above, but work rather more skilled or critical.	10
Ordinary work usually involving workers' inspection. Medium detail and contrast.	15
Fairly critical work, fairly small detail or poor contrast.	20
Skilled work, small detail or dark material.	30
Fine or critical work, very small detail, very poor contrast or very dark material.	50
Very fine exacting work.	100
Minute work.	200

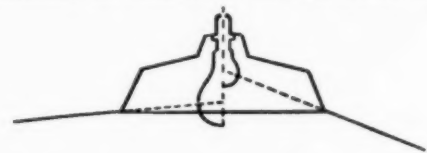
In addition, readings should be taken at selected working points with the lightmeter so placed that it measures the light on the work while the operative



is working. This will indicate whether full use is being made of the light or whether shadow is obscuring part of it.

Glare

Any direct light on the eye tends to reduce its sensitivity—thus reducing the power to see. Increasing the intensity of the light source may



therefore not improve matters unless care is taken to avoid glare either by correct positioning or by the use of correctly matched reflectors and lamps.

Walls and Ceilings

Certain surfaces and certain colours absorb light and therefore do not make the best of a light source; others reflect light and, so to speak, feed back on to the work a portion of the lighting which would otherwise be lost. Bright walls, moreover, have a good psychological effect which makes for contented—and therefore productive—operatives.

Shadows

Unnecessary shadows may seriously slow down work and also cause accidents. Lightmeter readings should always be taken under conditions exactly similar to those obtaining while work is going on. The operative himself may mask his work, an overhead crane or a heavily loaded conveyor belt may periodically obscure a light fitting, or a dust-laden atmosphere may reduce the designed lighting values.



For further information, get in touch with your Electricity Board or write direct to the Electrical Development Association, 2 Savoy Hill, London, W.C.2. Telephone: TEMple Bar 9434.

Excellent reference books are available on electricity and productivity (8/6 each or 9/- post free)—"Lighting in Industry" is an example.

E.D.A. also have available on free loan in the United Kingdom a series of films on the industrial uses of electricity including one on industrial lighting. Ask for a catalogue.

LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC

INSTRUMENTS AND MATERIALS

SEPTEMBER, 1959.

Vol. LX, No. 359

Test Blocks for Indentation Hardness Testing

By Mrs. J. G. Wood, B.Sc. (Eng.)

(Communication from the National Physical Laboratory)

The methods adopted at the N.P.L. for the production of standard hardness test blocks in the range 200 to 950 HV for the Vickers and Rockwell indentation hardness tests are described. Typical results from examination of the finished blocks demonstrate the excellent and consistent uniformity of hardness achieved.

STANDARD hardness test blocks provide the simplest and, under proper control, probably the most accurate means of verifying hardness testing machines. Such blocks, calibrated by the manufacturers or one of the standardising authorities, are commercially available for the three principal types of indentation hardness measurement, i.e., Vickers, Rockwell and Brinell, and for the full range of hardness required in industry. The standard test blocks provide the means for transferring to industry the standard of hardness maintained by the manufacturer or the standardising authority, and it will be evident that (a) the standard is essentially vested in the machine used for their calibration, and (b) the accuracy with which the standard is transferred through the medium of the test block, will be very much governed by the uniformity of hardness of the block, since the calibration must be based on a "sample test" of five—or at most ten—indentations for economic reasons.

In recent years, a close study has been made at the National Physical Laboratory of the problems associated with the establishment and maintenance of high grade standards for the Vickers (HV) and Rockwell (HRC and HRB) scales of hardness. A new deadweight machine for the HV scale, employing loads of 30, 50, 100 and 120 kgf., and exploiting new principles of load control during the indenting process, has been constructed¹, and a new deadweight machine for the HRC and HRB scales is at present being built. A critical examination of the performance of the deadweight Vickers machine has been made. The use of statistical techniques permitted separate evaluation of the effect of most of the factors which influence the measurement of hardness by such a machine, but it was not possible to separate test block variability, i.e., variability due to non-uniformity of hardness of the block, from machine variability, i.e., variability due to random errors in the indenting procedure. To establish the precision of the machine itself, it was therefore essential to use test blocks of the highest attainable uniformity of hardness. None of the commercial sources of blocks—and they were sought throughout the world—consistently provided the requisite degree of uniformity, and it was thus necessary to investigate at the Laboratory the possibility of making

the more uniform blocks needed. The techniques developed provided standard test blocks covering the range 200 to 950 HV which were uniform within 10 HV at the upper end of the scale and within 4 HV at the lower end. Furthermore, all the blocks of a batch prepared at one time showed the same degree of uniformity and had closely the same hardness, a point of considerable importance when an extended series of measurements involving some two hundred or more indentations has to be made at any required hardness level. These blocks have enabled a full appraisal of the accuracy of the machine to be made. It has also been established that they are uniformly hard to sufficient depth for use in the Rockwell scale of measurement.

The availability of highly uniform standard hardness test blocks offers considerable advantages for the inter-comparison of the standards of hardness maintained by manufacturers and standardising authorities throughout the world, and thus contributes substantially to the realisation of a uniform standard of hardness. It also improves the accuracy with which the standard can be transferred to industry on the basis of a "sample test" of indentations. It is believed, therefore, that the techniques which have been evolved at the Laboratory will be of interest and value to those concerned with the calibration of hardness standards, and a description of the techniques is given in this paper.

Material

In initial experiments to obtain blocks harder than 900 HV a plain carbon steel (1.1% carbon, 0.4% manganese) was used. These blocks are designated *V* in subsequent tables of results. In later work, when the method of production had been finalised and softer blocks were required, an alloy steel (1.0% carbon, 1.2% manganese, 0.6% chromium, 0.5% nickel, 0.4% tungsten, 0.2% vanadium, 0.2% silicon, 0.01% sulphur, 0.03% phosphorus) known as "Staytoform" was used. Blocks from this steel are designated *F*. Both steels were supplied in 2½ in. by ½ in. bar, which was cut into 2½ in. lengths to form blocks 2.5 in. by 1.75 in. by 0.4 in.

Preparation and Hardening

The top and bottom surfaces of the blocks were ground to ensure a clean run-off of the heating medium.

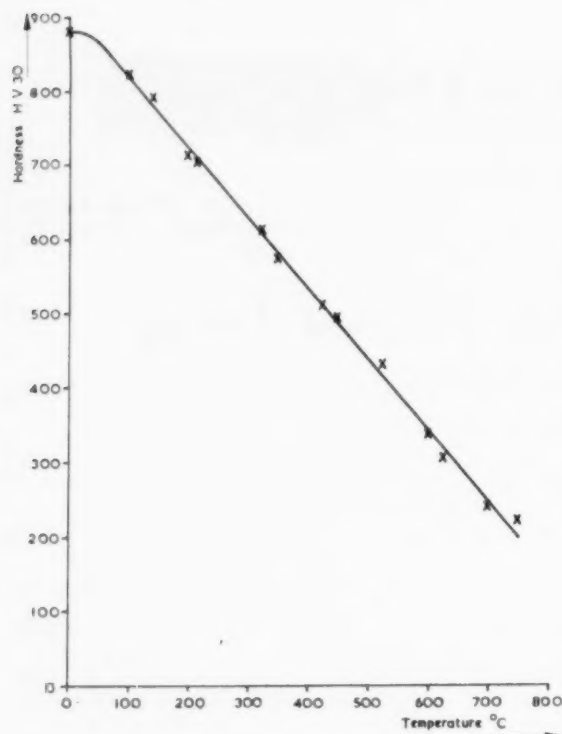


Fig. 1.—Effect of tempering Staytoform steel.

File cuts were made in the edges at the corners, so as to leave the surfaces unbroken, and a supporting wire wrapped round the periphery leaving the surfaces unimpeded.

The blocks were preheated to 450° C. and then immersed in a cyanide bath, containing 20–25% sodium cyanide, for seven minutes. This period was found to be sufficient for the block to attain a uniform temperature without decarbonisation of the surfaces. The temperature of the bath was maintained at 760°–780° C. for the plain carbon steel, and at 800°–820° C. for the alloy steel. It was found advisable to clean the bath before use with a regenerating salt, to prevent stray matter adhering to the surfaces and thus reducing the effectiveness of the quench over local areas.

The blocks were quenched individually in brine, composed of 1 lb. sodium chloride to 1 gal. of tap water, at room temperature. Temperature changes of the quench of several degrees did not affect the uniformity of hardness, but quenching in water without salt, or in oil, always produced blocks with local soft areas.

Tempering

To relieve partially the stresses induced by the quench, the hardened blocks were boiled in water for one hour, with a resultant reduction in hardness of the order of 60 HV. To obtain lower levels of hardness, the blocks were heated in an electric oven at temperatures ranging from 100°–750° C. The oven was rapidly raised to the required temperature, which was then maintained for three hours, after which it was allowed to cool slowly to room temperature.

The effect of the tempering temperature upon the hardness of the alloy steel is shown in Fig. 1, which

reveals a closely linear relationship over a wide temperature range, and a reduction of hardness of approximately 1 HV for a 1° C. rise in the tempering temperature. Blocks of this steel may therefore be produced at any desired hardness between 200 and 880 HV, within close limits, by a suitable choice of tempering temperature.

Mechanical Processing

The blocks were wet ground on a rotary surface grinder using a 38A60–G8VBE wheel which was regularly redressed during the grinding operation and set to advance the cut automatically by 0.0001 in. at each traverse. The blocks were turned over frequently to compensate for up to 0.005 in. initial bend produced during hardening, and to remove the distortion arising as stresses were relieved during grinding. As soon as both surfaces had cleaned up, a further 0.004 in. was removed from the working surface in 0.0001 in. cuts, and then 0.001 in. was removed by an automatic lapping process using Norbide 220 grit abrasive.

The surface was brought to a bright finish, with only fine abrasive scratches, by hand lapping with 2 μ m. diamond dust on a cast iron lap. A final mirror finish, with a centre line average roughness value of less than 0.5 μ m., was achieved by polishing on an 8 in. diameter wheel rotating at 400 r.p.m., the wheel being covered with thin rayon material moistened with ethylene glycol as lubricant and lightly impregnated with 1 μ m. diamond dust.

Recent limited experiments have shown that a dull lapped finish with a centre line average value of 3 μ m. can be obtained straight from grinding on a commercially available automatic lap using a fine abrasive in an oil base. This eliminates both lapping operations in the earlier procedure, and is particularly useful for the soft blocks which are not amenable to hand lapping.

Tests for Uniformity

The uniformity of hardness of blocks heat treated and machined by the methods described has been established in three ways.

- (1) By indenting the blocks on the NPL deadweight Vickers machine with a load of 30 kgf. and measuring the diagonals of the indentations on a specially designed measuring microscope.
- (2) By examining the metallurgical structure revealed by etching the polished surface of the block.

TABLE I.—UNIFORMITY OF BLOCKS RANGING FROM 220 TO 880 HV

Identification Number	Vickers Hardness (HV 30)	Number of Indentations in Set	Observed Range in Sets (HV units)
F.34	879	10	11
F.13	877	10	9
F.11	876	10	9
F.4	869	5	2
F.14	823	10	7
F.15	823	10	6
F.16	819	10	7
F.3	796	10	7
F.5	795	5	2
F.7	794	5	12
F.8	787	10	9
F.10	786	5	4
F.12	781	10	12
F.20	712	10	7
F.22	711	10	7
F.21	706	10	7
F.1	697	10	4
F.26	610	10	7
F.2	579	5	3
F.30	508	10	6
F.9	485	10	4
F.42	429	10	8
F.33	355	10	7
F.46	302	10	5
F.50	259	10	4
F.52	220	10	5

(3) By determining to what depth uniform hardness extends.

Results obtained by the first form of examination are tabulated in Tables I and II in terms of diamond pyramid hardness numbers, and those obtained by the third in Table III.

Surface Hardness

A representative number of *F* blocks within the range 220 to 880 HV were examined by making sets of ten Vickers indentations randomly disposed over the surface of each block, though in a few cases, during the initial work, sets of only five indentations were made. The ranges of hardness observed in each set of indentations are given in Table I. They show that the hardening technique developed gives consistent results, and the small range at the hard end of the scale, where most difficulty is experienced by industrial manufacturers, is particularly impressive. Although these values were obtained for a small sample of, at most, ten indentations, and therefore may not reveal the full variability of the hardness of a block, they do include additional variability due to small inherent errors in both the indenting machine and the measuring microscope.

A few *F* blocks which have been used extensively for experimental work provide a thorough examination of block variability. Many sets of ten indentations have been made on these blocks, and the minimum and maximum ranges observed, together with the average range, are tabulated for six blocks in Table II. As might be expected, the maximum ranges are larger than those displayed in Table I for a single set of ten indentations, but no appreciable lack of uniformity is revealed. In contrast with Table I, where the results are affected by the variability due to small sampling, the steadily decreasing average range shown in Table II confirms the generally accepted view that uniformity is improved by tempering.

The overall range of a *V* type block was revealed in an experiment in which nineteen sets of five indentations were made over the surface of the block, giving average, minimum and maximum ranges of 6, 2 and 11 HV respectively. These indentations were all made under the same conditions and the total range observed for this very hard block of 950 HV was only 14 HV.

Surface Structure

For a metallurgical examination of the hardened structure, a particularly fine polish was obtained on the test surface, which was etched in a 2% nital solution and then examined under a high power microscope. It became evident that a block of uniform hardness showed only martensitic structure, and that the presence of areas of troostite gave rise to inferior uniformity. This fact was demonstrated by the correlation between measurements of 35 indentations uniformly spaced over the surface of a *V* block and the structure surrounding each

TABLE III.—UNIFORMITY OF HARDNESS AT LEVELS THROUGH A BLOCK

Depth (in.)	0.005	0.008	0.013	0.023	0.033
Hardness (HV30)	931	932	926	927	910
Range (HV units)	10	12	7	8	14

indentation, which was revealed by etching to a limited extent, so that the troostite areas were appreciably affected whilst the martensite was only lightly attacked. The average hardness of the troostite areas was 924 HV with a range from 902 to 939 HV, while the average hardness of the martensite areas was 943 HV with a range from 940 to 949 HV. It should be noted that these ranges do not overlap, and the results emphasise the importance of fully hardening the steel in order to obtain the uniformity conferred by a martensitic structure. These distinct ranges also show the danger of applying statistical techniques to hardness values, which are unlikely to be normally distributed if more than one structure is present.

Depth of Hardness

The uniformity of hardness at different depths was obtained for a *V* block by indenting and etching at five levels through the block. The first level was the normal working surface, which is between 0.005 in. and 0.010 in. below the original surface, and further amounts of 0.003, 0.005, 0.010 and 0.010 in. were ground away in turn, the surface being lapped and polished at each stage. The average hardness measured and the range revealed by five indentations at each level are set out in Table III, and show that the first appreciable decrease in hardness occurred at a depth of about 0.030 in. below the usual working surface. This surface was the first to reveal traces of troostite, and was therefore considered to be the limiting depth for full hardening. From the constitution of the alloy steel, a greater depth of hardening may be expected from the *F* blocks, and it is therefore concluded that both types of blocks provide sufficient depth of uniform hardness for the Vickers and Rockwell tests envisaged at the Laboratory.

Conclusions

The three most important factors affecting the heat treatment process adopted are that the salt bath shall be free from sludge, that the temperature of the blocks shall be within the prescribed limits when quenched, and that the quench shall be rapid and uniform. The latter requirement was only fulfilled by the use of a brine bath.

During mechanical processing, the grinding conditions are particularly critical: localised overheating of the surface by using a wheel of incorrect grit size or bonding, or by using too heavy a cut at any stage of the surfacing, may ruin an otherwise satisfactory block. It was for this reason that the removal of at least 0.001 in. of metal by mechanical lapping was introduced, since even the lightest grinding produces some local overheating.

Flatness of the lower surface of the block is more important than is sometimes realised. A rocking block will enlarge Vickers indentations, while a block which flexes under load will not permit true depth measurement in the Rockwell test. Attention must therefore be paid to the grinding of this surface.

A mirror finish, which is readily obtained with diamond dust polishing, even with soft blocks, is considered to be essential if precision is to be obtained from the Vickers test. The high reflectivity of the polished

TABLE II.—UNIFORMITY OF BLOCKS SUBJECTED TO A DETAILED EXAMINATION

Identification Number	Vickers Hardness (HV30)	Number of Sets of 10 Indentations	Average Range (HV units)	Minimum Range in a set (HV units)	Maximum Range in a set (HV units)
F.34	879	16	8	4	12
F.13	877	16	8	4	12
F.11	876	7	9	2	16
F.1	697	6	5	2	10
F.9	485	14	5	3	8
F.52	220	10	2	1.5	3

surface increases the contrast between the indentation and the surrounding surface of the block seen in the measuring microscope, and, in addition, the corners of the indentations are not marred by scratches or grinding marks. The mirror finish also provides a surface suitable for etching, and this technique provides a rapid means of revealing the presence of soft spots, which is particularly useful during periods of experimental hardening, or if there is doubt concerning the quality of a block.

Hardness test blocks are now made by the heat treatment and finishing methods described, with consistently successful results, and the uniform blocks so provided have enabled a critical appraisal to be made of both the standard deadweight machine and the prototype measuring microscope which were built for the Vickers test at the N.P.L. In addition, they have enabled a critical examination to be made, in both Vickers and

Rockwell tests, of the influence of small variations in the indenting cycle, and confirmed, what had been earlier suspected, that the cycle must be precisely defined.

Acknowledgments

The author wishes to acknowledge the advice of Mr. H. Bull, of Messrs. Brown Bayley Steels, Ltd., who recommended the use of "Staytoform" Steel, and the advice and assistance of Mr. R. S. Marriner throughout this work. The work described above has been carried out as part of the research programme of the National Physical Laboratory, and this paper is published by permission of the Director of the Laboratory.

REFERENCE

- 1 A New Standard Hardness Testing Machine, Marriner R. S. and Mason, F. C. P. *Machinery*, 1957, **91**, 1225; *Machine Shop Magazine*, 1958, **19**, 31; *Metallurgia*, 1958, **57**, 47; and *Engineering*, 1958, **155**, 539.

A Simple Flow-Through Cell for use with the Unicam SP.600 Spectrophotometer

By C. J. Keattch,* A.R.I.C. and P. W. Wright

H. J. Enthoven & Sons, Ltd., Rotherhithe, London, S.E.16

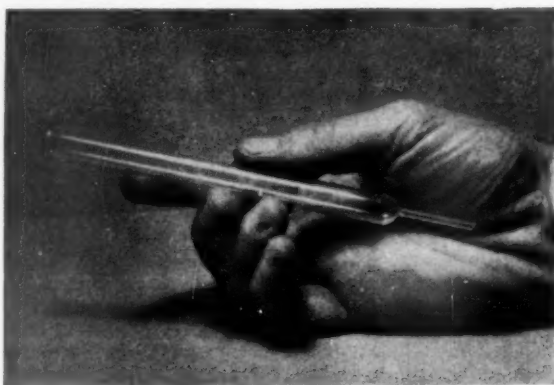
THE Unicam SP.600 spectrophotometer, with its accurate method of wavelength selection and general extreme robustness, is an excellent instrument for colorimetric determinations on a routine basis. In repetition work, where the emphasis is on speed as well as accuracy, one of the instrument's few disadvantages becomes manifest, namely the lack of a commercially available flow-through cell.

It was an attempt to overcome this disadvantage that has resulted in the development of the simple flow-through cell described in this paper. The cell is used with the test-tube holder supplied by the manufacturers, but overcomes the drawbacks that accompany the orthodox use of this attachment; namely that the degree of accuracy attainable depends upon the quality and matching of the test-tubes used. Also that there can be a variance of up to 8-10% transmission by merely rotating the test-tube in the holder.

The determination for which this cell was originally designed is the well-known method for determining copper using hydrobromic acid-bromine as the colour developing reagent. In this particular instance it has the added advantage that the corrosive hydrobromic acid fumes are excluded from the main body of the instrument—a criticism of the method quoted by many potential users. The use of this cell is not, of course, limited to this determination alone.

Construction and Use

The flow-through cell needs very little skill to make, and consists of a 1-in. length of 3 mm. o.d. tubing blown on to the bottom end of a Pyrex test-tube 6 in. \times $\frac{1}{2}$ in.† A length of polythene tubing is attached to the end of the glass tube. The cell is then assembled in the test-tube holder by threading the polythene tubing through one of the holes in the base of the holder and passing it underneath the instrument. It was found that this could best be accomplished if the spectrophoto-



meter were raised slightly. This was achieved by removing the four rubber feet on the base of the instrument and replacing them by rubber door stops approximately 1 in. high \times 1 in. diameter. The end of the polythene tubing is connected to a vacuum line incorporating a glass stop-cock, to operate the emptying of the cell, and a reservoir to trap spent solution.

To use the cell it is merely rinsed through two or three times with the solution, filled to a convenient height and a reading taken in the usual way. The instrument is set in the same manner. It was found that the light excluding cover supplied by the manufacturers was not necessary, completely reproducible results being obtained over a wide range of concentrations. These cells have been in routine use in our laboratories for more than a year with complete success.

Acknowledgment

This work was carried out in the Laboratories of H. J. Enthoven & Sons, Ltd., Rotherhithe, London, and thanks are due to the Directors for permission to publish this paper.

* Now at the Mond Nickel Co., Ltd., Acton, London N.W. 10.

† Also obtainable from R. & L. Slaughter, Mawney Road, Romford, Essex.

Metallurgy of the New Metals

New Laboratory for Nuclear Metals Inc.

THE new plant of Nuclear Metals, Inc., at Concord, Massachusetts, is considered to be one of the most completely equipped metallurgical facilities in the U.S.A. Complete equipment is available to support research and development programmes extending from fundamental studies to fabrication and prototype manufacturing of large and complex products for the nuclear, aircraft, missile and electronics fields. A feature of the equipment is the extensive hooding and ventilation which permits handling toxic, radioactive, and reactive materials such as beryllium, uranium, enriched-uranium, thorium, zirconium, and yttrium. The plant is especially well equipped for high temperature work.

Laboratory Building

An H-shaped glass and brick structure was the best solution to the various parameters assigned to the architect, J. Q. Adams, in the design of the new research and development laboratory. These parameters included a need for maximum natural light for intricate metal working and fabrication activities, the use of very large amounts of electric power in the building, and unusual ventilation requirements.

Long corridors in the building are softened by the use of alternating colours, and light pastel shades are used throughout the 86,000 sq. ft. building. The entire building is developed on four foot modular systems to allow ease of expansion or remodelling of the interior. All walls except those of the main corridor are easily movable, and the modular system permits rearrangement of the whole interior at minimum cost. The building is in three main parts. One leg of the "H" contains reception and conference rooms, administrative, accounting and library facilities, as well as laboratories. The cross bar of the "H" houses boiler room, electric switch rooms, telephone switchboard room and emergency generator. From this central location, the electric lines are carried to the far ends of the building. The central portion also contains lavatory and shower rooms, lockers, first-aid rooms, and a cafeteria.

The other leg of the "H" contains the melting and fabrication section of the building. This section contains the machine shop, foundry, fabrication, welding, sheet metal, and carpentry shops, together with a very large truck entrance for shipping and receiving. The 26 ft. ceiling in this area provides ample height for vertical presses, cranes and other apparatus.

The equipment is described below according to the following functional classifications: (1) material preparation and machining; (2) fabrication and prototype production; (3) material treatment; (4) physical, mechanical, and chemical measurement, testing, and evaluation; (5) other.

Material Preparation

A research or development effort usually begins with the production of suitable quantities of pure metals, alloys, ceramics, or cermets. Foundry equipment for melting in vacuum, atmosphere, or air, includes a 100 kW. motor generator induction unit; a number of 40 kW.



A 2 in. diameter billet is ready for insertion in the liner of the 300-ton extrusion press.

and 20 kW. high frequency induction units; quartz and internally cooled Micarta tube bottom-pour vacuum furnaces; and facilities for making moulds and crucibles of materials such as beryllium oxide, zircon, magnesium zirconate, and high purity graphite. Melting capacity is up to 300 lb. of uranium or 200 lb. of steel in crucibles up to 9 in. internal diameter, and large vacuum systems permit melting up to 3,600° F. (1,982° C.) at vacuums as low as 25 microns.

Under construction is a cold-crucible arc melting unit capable of vacuum melting castings up to 8 in. diameter and 30 in. long; the electrodes may be either consumable or non-consumable; and power will be supplied by a 6,000 A. D.C. rectifier source.

Hooded hydraulic presses ranging in capacity up to 270 tons, along with a variety of pulverizers, graders, blenders, ball mills, furnaces, and hot compacting units, are available for powder metallurgy preparation of metals, cermets, and ceramics. For large sizes and refractory metals, the 1,000 ton extrusion press is adapted for cold and hot compacting.

A large machine shop, with each machine hooded or ventilated to allow machining of toxic, reactive, and radioactive materials, permits intricate shaping of a wide variety of materials. Included in the equipment are some twenty-five lathes from 10 in. to 18 in. capacity; milling and shaping machines; surface, cylindrical and thread grinders; large radial drill and ordinary drill presses; and power hacksaws and abrasive cut-off wheels.

Complete welding facilities, including a dry box system, plus a sheet metal shop and a carpenter shop provide additional means for material preparation.

Fabrication and Treatment

Nuclear Metals possesses a wide variety of metal-working equipment for hot and cold forming of materials. The 1,000 ton horizontal extrusion press is believed to



A battery of furnaces having temperature ranges up to 2,600° F. (1,427° C.) is arranged to service conveniently both the 300 ton and 1,000 ton extrusion presses.

be the largest extrusion press in the world devoted primarily to research and development work. Other extrusion equipment includes a 300 ton horizontal press; a 100 ton fast-acting vertical press; a large variety of extrusion containers ranging from 0.900 in. to 8 in. diameter; and a battery of furnaces capable of reaching temperatures up to 2,600° F. (1,427° C.).

Other metal-working equipment includes: an 8 in. rolling mill; three draw benches ranging in capacity from 3,000 to 15,000 lb.; four swaging machines ranging in capacity from 0.010 in. diameter wire to 3½ in. diameter tubes; and a centreless belt grinder.

Through excellent working arrangements with one of its parent companies and other large mills and fabricating concerns, Nuclear Metals is able to augment its own extensive metal-working facilities by renting time on larger equipment, including rolling mills, extrusion presses, forge presses, hammers, and draw benches.

A wide variety of equipment is available for the thermal and chemical treatment of metals and materials. Furnaces of all sizes and shapes are available, including induction heated units and resistance furnaces heated by Globar, nickel-chromium, platinum and molybdenum elements. Temperatures of over 3,000° F. (1,649° C.) in a high vacuum, or in hydrogen or other atmosphere, can be obtained. Salt and lead baths permit rapid heating of billets and samples, and three 20 ft. long vacuum annealing furnaces are available for bright annealing Zircaloy and other reactive metals: zone-melting equipment is also available.

A battery of pickling and etching tanks can handle rods, tubes, and shapes up to 20 ft. in length, and surface treatment of materials can be carried out by electroplating, anodizing, and sputtering equipment.

Measurement, Testing and Evaluation

A large quantity of equipment is installed for making measurements to characterise materials. Equipment for physical measurements permits determination of shape, size, density, electrical properties, thermal conductivity, magnetic properties, and low temperature measurements: a dilatometer is available for operation in controlled

atmospheres. For X-ray diffraction studies, a Norelco diffractometer and Picker diffraction units are in use with several types of X-ray cameras, including a Unicam single crystal goniometer; a precision back-reflection camera; and a high temperature vacuum camera capable of operating at temperatures exceeding 1,300° C.

Straightforward determination of mechanical properties can be made on the 20,000 lb. tensile testing machine and the five types of hardness testing machine, which include the Rockwell, Brinell, Vickers and Tukon micro-hardness instruments. For assessing the high temperature mechanical properties there are creep and stress-rupture units capable of operation in controlled atmospheres at 2,300° F. (1,260° C.). Other equipment concerned with less commonly quoted mechanical properties include some devices for the determination of elastic properties; internal friction measuring devices; and optical comparators. Non-destructive testing for soundness can be carried out by ultrasonic, magnetic, eddy current and radiographic methods, a 150 kV. unit being provided for the last-named.

The metallographic laboratory is fully equipped with the usual specimen preparation equipment, including an electropolishing unit. The two Bausch and Lomb metallographs are supplemented by a number of bench microscopes, whilst low power work is catered for by macro-cameras. A comprehensive range of photographic processing equipment completes the equipment of this laboratory.

A chemical analysis laboratory is maintained for the determination of constituents and impurities, and for devising methods of analysis of new material systems. Included in the equipment are a micro-Kjeldhal distillation unit, an Everbach Dyna-catch, a Fisher electro-analyser, Galvanek-Morrison and Farrand fluorimeters, a Larabee titrator, and Beckman pH meters and spectrophotometers (including a flame photometer attachment). This equipment, together with a vacuum fusion apparatus, a spectrographic laboratory and X-ray fluorescence equipment, permit use of a wide variety of analytical techniques.

Other Equipment and Facilities

The work of the laboratories is supported by a glass

(Continued on page 124)



Removing experimental clad reactor fuel element material from furnace preparatory to insertion in the rolling mill.

The Analysis of Nickel

Part I—Chemical Methods

By T. R. Andrew and C. H. R. Gentry

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A critical survey is given of published methods for the chemical determination of the alloying constituents and impurities in electronic nickel. On the basis of a re-examination of the published methods and additional investigations, specific methods for each element have been formulated. Detailed instructions are provided for carrying out the several methods with statements of the time required and the precision attainable under routine conditions. Several of the methods, which are mainly photometric, are original, and all should be applicable to other non-ferrous metals in addition to nickel.

(continued from page 72 of the August issue)

Magnesium

The difficulty of determining the important element, magnesium, in electronic nickel is indicated by the fact that the A.S.T.M.^{2,3} do not include a method for it in their nickel analysis procedures. A procedure advocated by Inco¹ requires a 5 g. sample and finishes gravimetrically as magnesium pyrophosphate after several separation stages. Some fifteen years ago, in this Laboratory, a chance observation led to the development of a method in which the bulk of the nickel, together with aluminium, iron, titanium, etc., is separated from magnesium by precipitation as nickel hexamminopercchlorate, and the magnesium is subsequently determined gravimetrically as the 8-hydroxyquinolate.

Since this procedure has given very satisfactory results over a number of years, and is simpler in operation than the Inco procedure, details are provided here:—

5 g. nickel is dissolved in 50 ml. nitric acid (1 : 1) and evaporated to fumes with 40 ml. perchloric acid (S.G. 1.7). After cooling, 150 ml. water is added and ammonium hydroxide (1 : 1) until alkaline with a 5 ml. excess. The solution is allowed to stand on a steam bath for $\frac{1}{2}$ –1 hour, and the crystalline precipitate of nickel hexamminopercchlorate filtered off in a large sintered glass funnel and washed with dilute ammonium hydroxide (3 : 97). The filtrate is evaporated to 100–120 ml. and 1 g. tartaric acid added, followed by 10 ml. ammonium hydroxide (1 : 1), 1 g. sodium cyanide and 1 g. sodium sulphate.

The cold solution is gassed with hydrogen sulphide and any precipitate filtered off. Magnesium is precipitated in the filtrate by heating to 70° C. and adding 10 ml. ammonium hydroxide (1 : 1) followed by excess 5% alcoholic 8-hydroxyquinoline. The precipitate is filtered on a small sintered glass crucible (porosity 3) and weighed after drying at 100°–105° C.

This procedure suffered only from the need to take a large sample weight (5 g.) and could not meet the requirement of applicability to single valve cathodes weighing perhaps only 10–50 mg. For this reason, many other methods have been tried by the authors, and every new procedure published during the last ten years, of which they are aware, dealing with the determination of small amounts of magnesium, whether in nickel or not, has been examined. In this work, it has been an article of faith that a satisfactory method should be based on com-

plete recovery of magnesium, and should not rely on empirical calibration by means of standards.

The Titan yellow procedure of Yokosuka⁶⁶ on a 2 g. sample after mercury cathode electrolysis was rejected as unwieldy. Luke and Campbell's⁶⁷ method, based on extraction of the 8-hydroxyquinolate in the presence of butyl cellosolve after preliminary separations, and Luke's⁶⁸ subsequent modification were not satisfactory in the authors' hands, principally because they required very precise control of the pH of extraction. The proposal of Umland and Hoffman⁶⁹ to separate magnesium from other metals by extraction with 8-hydroxyquinoline and chloroform at pH 5, followed by addition of *n*-butylamine and extraction with 8-hydroxyquinoline at pH 11, fails because of loss of 15–30% of the magnesium during the extraction at pH 5. Similar objections mar the work of Noddack, Eckert and Reidel⁷⁰ and of Pohl,⁷¹ who separate nickel by extraction with diethyldithiocarbamic acid and chloroform, or by 8-hydroxyquinoline followed by sodium diethyldithiocarbamate and chloroform. In both these techniques, some 10–20% of the magnesium present is lost during this preliminary separation, the exact amount being dependent on the vigour of shaking during the extractions.

It seemed probable that this loss of magnesium would be common to all procedures in which several extractions are made to separate the heavy metals; and the authors accordingly turned their attention to the possibility of determining magnesium in nickel without such separations. Although criticisms have been levelled at the Eriochrome black T method for magnesium, notably in respect of fading of the colour and the high blank obtained, it appeared worthwhile to examine this reagent and the closely related Eriochrome greys BL and SGL suggested by Knop.⁷²

In view of the use, by Pribil⁷³, of potassium cyanide to mask interference of nickel in titrations with ethylenediaminetetra-acetic acid, using Eriochrome black T as indicator, consideration was given to the use of this method to overcome the need for the preliminary separations of heavy metals. Preliminary experiment showed promise and enabled a choice to be made between the Eriochrome dyestuffs.

Eriochrome black T is used generally as a 0.05% solution in alcohol and is only slightly soluble in water; the specimen used in the authors' work also appeared to

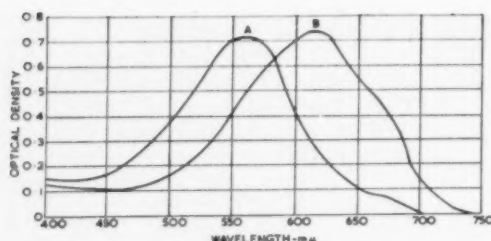


Fig. 1a.—Absorption spectra: Eriochrome grey 3BL 200% using final conditions (1 cm. cell). A—excess magnesium (100 $\mu\text{g.}$); B—no magnesium.

contain about 20% alcohol-insoluble material. Eriochrome grey 3BL 200%, however, is readily soluble in water, and this was felt to be a real, though slight, advantage. Consideration of the absorption spectra of the two dyestuffs, in the presence and absence of excess magnesium (Fig. 1), showed that Eriochrome grey gave sharper peaks than Eriochrome black, and that the optical density of the blank at equal concentrations of dyestuff, was less in the region in which the magnesium complex absorbed. It was clear from an examination of these spectra that, in both cases, measurement could be made either at 520 $m\mu$, measuring the optical density of the magnesium complex against the dyestuff, or at 620 $m\mu$, measuring the residual dyestuff. The results of work on this are plotted in Fig. 2, which shows clearly that at 620 $m\mu$ both dyestuffs give curved graphs, that of Eriochrome black T being the more pronounced; at 520 $m\mu$, Eriochrome grey 3BL 200% gives a linear calibration up to 35 $\mu\text{g.}$ magnesium/50 ml., while Eriochrome black T is non-linear but shows only a slight curve up to about 20 $\mu\text{g.}$ magnesium/50 ml. It must be conceded that Eriochrome black T is the more sensitive reagent, but, in order to cover the whole range of magnesium levels, with this extra sensitivity, it would be necessary to increase the amount of dyestuff added, and it should not be forgotten then that the dyestuff solution is used as the comparison solution.

Accordingly, Eriochrome grey 3BL was chosen and a study made of the interferences and methods for overcoming them. The procedure used for this study was simple: to the solution containing magnesium was added the suspected interference, followed by ammonia-ammonium chloride buffer, potassium cyanide solution and the dyestuff. The optical density at 520 $m\mu$ was then compared with the value obtained in the absence of the interference. The results of this study are given in Table III. It was found that, while the sensitivity of the procedure was dependent to a small extent on the amount of potassium cyanide added, presumably due to slight change in pH, the presence of nickel was without effect, provided that sufficient cyanide were present to complex it.

TABLE III.—EFFECT OF OTHER IONS ON MAGNESIUM DETERMINATION

Element	Amount Added ($\mu\text{g.}$)	Magnesium Equivalent ($\mu\text{g.}$)
Aluminium	25	ca 0.1
Calcium	25	2
Cobalt	2,500	Nil (see below)
Manganese	1,000	Nil (see below)
Nickel	25,000	Nil
Titanium	25	0.5
Tungsten	1,000	Nil

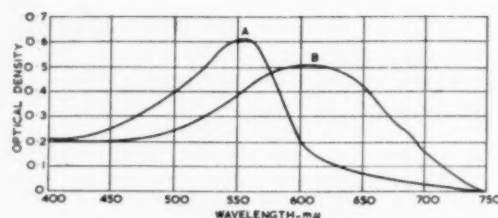


Fig. 1b.—Absorption spectra: Eriochrome black T at same concentration. A—excess magnesium (100 $\mu\text{g.}$); B—no magnesium.

Manganese and cobalt both give rise to interferences, but these can be eliminated by heating the solution after the addition of cyanide. In the case of manganese it is sufficient to bring the solution just to boiling, while full conversion of cobalt to cobaltcyanide needs a boiling period of 1 minute. Unfortunately, if the manganese content exceeds about 0.2%, a precipitate (presumably manganese cobaltcyanide) can form during this period, and must be removed before attempting to measure the optical density of the solution. It has been found most convenient to complete the colour formation and dilution of the solution and then, if necessary, to centrifuge a portion before measurement. No loss of magnesium has been found using this technique.

Two other points are worthy of attention. Firstly, the use of quartz vessels is often recommended, but negligible pick-up of magnesium has been found from ordinary unscratched resistance-glass ware after thorough cleaning. Secondly, the reagent shows a slight fall in sensitivity on standing, possibly due to photochemical decomposition, since this can be reduced to very small proportions if the reagent is kept in the dark. In practice, one can prepare a fresh solution and use it within a few hours, keeping it protected from strong sunlight.

The procedure in its final form, which has given excellent results and good agreement with classical procedures on a larger scale, is as follows:

Dissolve 25 mg. sample in 2 ml. nitric acid (1:1), add 2 ml. hydrochloric acid (1:1) and evaporate just to

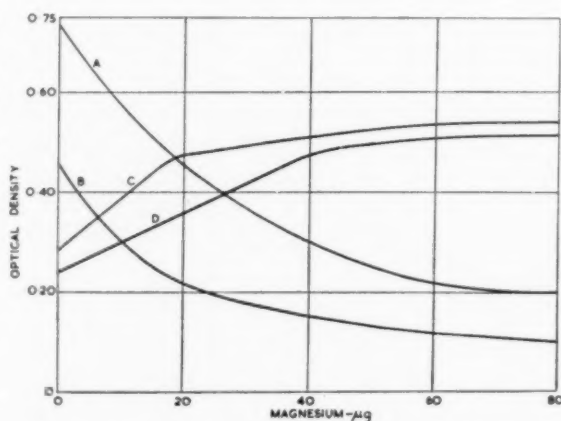


Fig. 2.—Calibration curves. A—620 $m\mu$., Eriochrome grey; B—620 $m\mu$., Eriochrome black; C—520 $m\mu$., Eriochrome black; D—520 $m\mu$., Eriochrome grey.

dryness (do not bake). Dissolve the residue in 10 ml. water and add 10 ml. buffer solution (60 ml. ammonium hydroxide, 10 g. ammonium chloride in 1 litre) and 10 ml. freshly prepared 2.5% potassium cyanide. Heat to boiling and boil for 1 minute. Cool rapidly and transfer to a 50 ml. graduated flask. Add 2.0 ml. 0.05% Eriochrome grey 3BL 200% and dilute to mark. Mix well and centrifuge if any turbidity is present.

Prepare a blank solution in the same way, omitting the sample. Measure the optical density of the solution against the blank at 520 m μ , using 4 cm. cells. If a filter instrument is used, a filament lamp, H503 and Ilford filters are suitable, although in this case, due to the wide band width of the filter, the calibration graph will show a slight curve. The range of the method is up to 0.14% magnesium.

A calibration graph is prepared using magnesium metal. It is not necessary to boil or to add nickel in the preparation of this calibration graph.

The procedure is extremely rapid, and six samples may be analysed in under two hours. On the Mond Nickel Company's standard samples, duplicate analyses carried out at different times showed a maximum deviation from the mean value of 0.002% magnesium. The coefficient of variation in the range of 0.02–0.25% magnesium was $\pm 2.3\%$.

Manganese

Volumetric methods for determining manganese in nickel have been described by Inco¹ and A.S.T.M.² Two methods are given, a bismuthate oxidation and ferrous ammonium sulphate titration after a preliminary separation with ammonia-ammonium persulphate, and a persulphate oxidation followed by a sodium arsenite titration. Apart from the time required, the main objection to these methods is the fact that a sample weight of several grams is required.

Several papers^{5,74,75,76} describe the photometric determination of manganese in nickel compounds or plating baths after oxidation, either with persulphate in the presence of a silver catalyst, or with potassium periodate. The A.S.T.M.³ have adopted a periodate oxidation for electronic nickel, and B.S.I.⁷⁷ have also used this method for the absorptiometric determination of manganese in steels, including high-nickel steels. Small amounts of manganese ($<0.01\%$) in nickel have been determined by Yokosuka⁷⁸ by co-precipitation with hydrous ferric oxide, followed by spectrophotometric measurement after oxidation with ammonium persulphate. A different approach has been adopted by Eckert,⁷⁹ who removes the nickel by extraction with zinc diethyldithiocarbamate into chloroform, and then determines the manganese with formaldoxime. He claims a mean error of $\pm 5\%$ in the range 0.005–0.1% manganese.

The present authors prefer a photometric method utilising the permanganate colour rather than the formaldoxime method of Eckert, which is more time-consuming and—although useful for small amounts of manganese—is comparatively lacking in precision at, say, the 0.05% level. Of the two methods of oxidising manganese to permanganate, experience over a number of years in a routine laboratory analysing a wide range of materials has strengthened a preference for the periodate method, which does not suffer from the fading and the difficulties which sometimes bedevil the persulphate

method in the hands of semi-skilled operators. The periodate method is very simple in operation and no prior separations are required. In the A.S.T.M. versions, the fuming with perchloric acid, filtration of tungstic oxide, silica, etc., and the removal of copper are unnecessary preliminary steps which can only detract from the accuracy of the final answer. For many years, manganese has been determined in this Laboratory by periodate oxidation in the presence of nitric, sulphuric and phosphoric acids; in the past five years, the details have been slightly modified to accord with those given in the British Standard method for determining manganese in steels. This step was taken in the interest of standardisation, and since it is obviously of value to utilise a thoroughly tested method for as wide a range of materials as possible.

As applied to nickel analysis, the procedure is as follows:—

Digest 0.25 g. sample in 20 ml. nitric acid (1 : 1) and 35 ml. phosphoric-sulphuric acid (3 : 3 : 14) until solvent action ceases, and then boil for 2 minutes. Add 10 ml. potassium periodate solution (5% in nitric acid (1 : 5)) and boil gently for 5 minutes. Leave on the sand bath for 30 minutes, if the expected manganese content is small. Cool, transfer to a 100 ml. graduated flask and dilute to the mark. Adjust the temperature to $20 \pm 2^\circ \text{C}$. Fill a 4 cm. cell with the solution (colour solution). To the solution remaining in the flask, add a 1% solution of sodium nitrite dropwise until the permanganate colour is discharged and 1 drop excess. Fill a second 4 cm. cell with this compensating solution. Measure the optical density of the colour solution, using the compensating solution as the blank. Suitable conditions are either an absorptiometer with a mercury vapour lamp and Ilford 605 with H503 filters or a spectrophotometer at 520 m μ . The range of the method with the absorptiometer is up to 0.2% manganese.

For calibrating the method, a solution obtained by weighing out AnalaR potassium permanganate and dissolving in a known volume of water may be used. Suitable aliquots are taken and carried through the procedure. It is not necessary to reduce this permanganate solution before use, or to add pure nickel to it; it is, however, important that the acid concentrations at the oxidation stage are adhered to.

It is necessary in the procedure to use pure water, free of reducing agents. The supply should be tested by adding to 500 ml. of it a few drops of dilute sulphuric acid, and two drops of 0.1N potassium permanganate. On heating to boiling, the pink colour should not entirely disappear. Some warning might also be given concerning the source of potassium periodate. No difficulties have ever been encountered with British material (AnalaR), but certain Continental supplies of analytical-grade reagent have been stated to give fading and low results.⁸⁰ The cause of this is not known to the authors.

Provided that the acid concentrations are strictly adhered to, the basic procedure is readily amenable to permit smaller samples to be analysed, greater precision to be attained at the lower manganese contents, or manganese contents higher than 0.2% to be determined. Thus, by using a final volume of 20 ml. and 4 cm. micro-cells, it is possible to analyse samples as small as 10 mg. with little loss of precision.

The method is not interfered with by any of the other

constituents of electronic nickel, as would be expected from the fact that it is applicable to a very wide range of steels. In particular, chromium does not interfere, and tungsten presents no difficulty since it is kept in solution by the phosphoric acid. The method is fast, and batches of samples can be analysed in a time equivalent to ten minutes per determination. On the basis of replicate analyses of the Mond Nickel Company's standard samples, the precision of the method was such that the average deviation from the mean values was 0.001% manganese over the range covered by these samples.

Phosphorus

Electronic nickel contains only trace amounts of phosphorus, at a level where the photometric methods devised for steel analysis were worth considering. It was felt preferable to avoid molybdenum blue techniques in view of possible interference by silicon, and the phospho-vanado-molybdate extraction of Elwell and Wilson,⁸¹ of which this Laboratory had considerable experience, was therefore tried.

It was found possible to simplify considerably the published procedure, in view of the restricted amount of alloying constituents in nickel, and the procedure eventually adopted is given below:—

Dissolve 0.25 g. sample in 10 ml. nitric acid (1 : 1) in a 100 ml. conical flask. Boil to expel brown fumes, add 5 ml. water and filter (if necessary) through a small 540 filter paper into a second 100 ml. conical flask. Wash with 10 ml. warm water and discard any residue. Heat to about 80°C. and add 25 ml. ammonium vanadate-molybdate reagent (see below). Cool to 20°C. and transfer to a 100 ml. separating funnel, dilute to about 80 ml. and add, from a burette, 12 ml. isoamyl alcohol (milk testing quality). Shake vigorously for 3 minutes, allow to stand for 5 minutes, run off lower layer and reject. Transfer the organic layer to a 4 cm. absorption cell through a dry filter paper, and record the optical density at 425 mμ. A blank determination should be made, preferably on phosphorus-free nickel. The range of the method is up to 0.015% phosphorus.

The ammonium vanadate-molybdate reagent is prepared as follows. Dissolve 1 g. ammonium vanadate in 300 ml. water. Add slowly 140 ml. concentrated nitric acid, followed by 40 g. ammonium molybdate in 400 ml. water. Dilute to 1 litre.

A calibration graph is prepared in a similar fashion from phosphorus-free nickel to which is added 0.01, 0.02, 0.03, 0.04 mg. phosphorus as a solution of potassium dihydrogen phosphate.

A batch of six samples can be analysed in about an hour and a half. The reproducibility of the method has been tested only on synthetic solutions, since a range of nickel samples containing phosphorus was not available. The highest phosphorus content found in a nickel sample has been 0.001%, at which level duplicates agree to within $\pm 0.0001\%$ phosphorus. (To be concluded)

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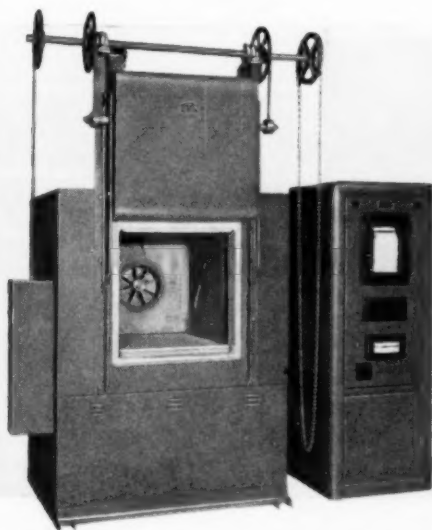
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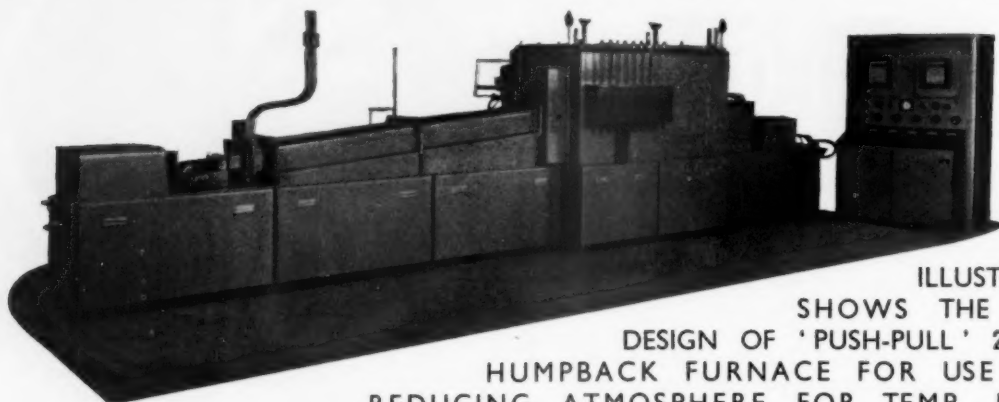
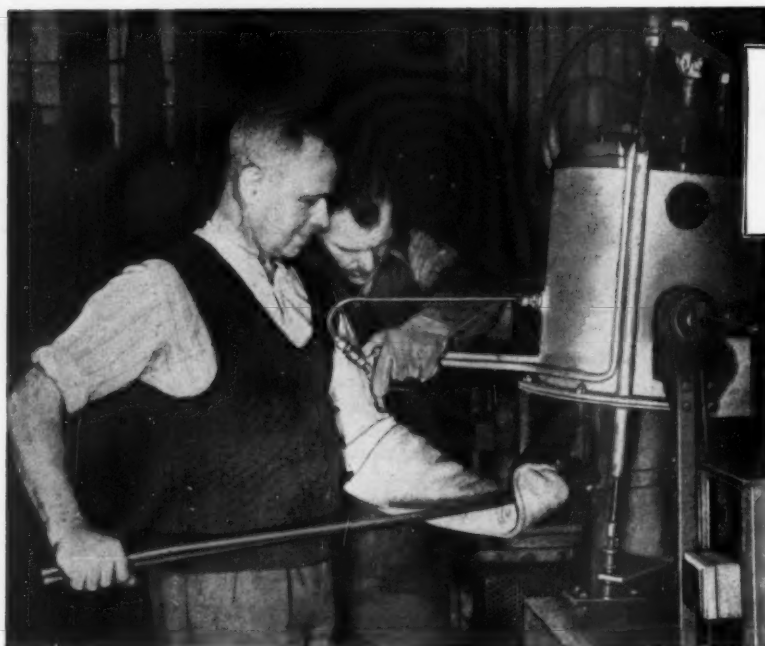


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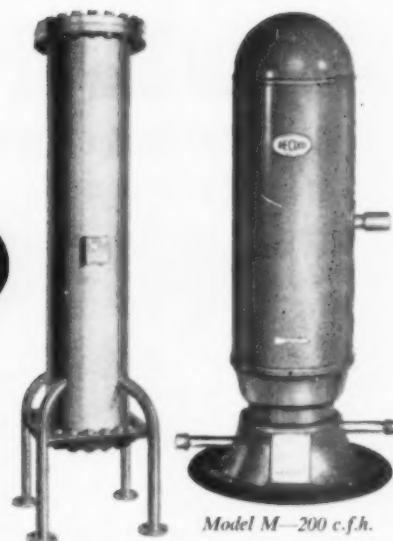
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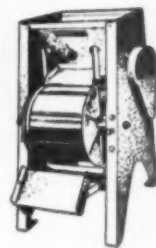
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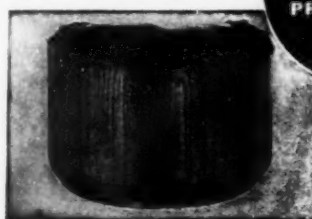


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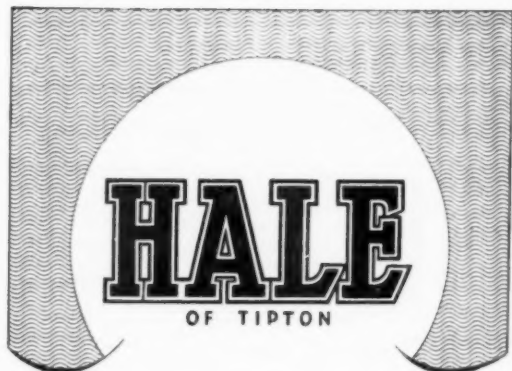
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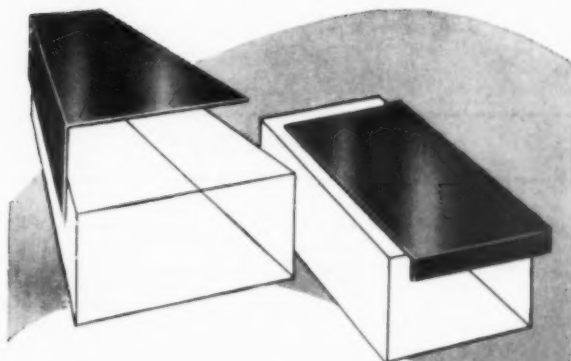
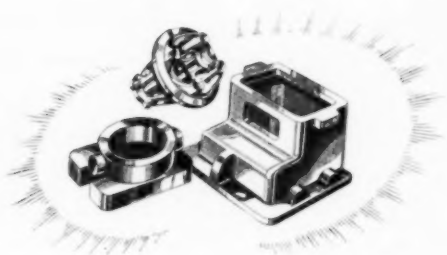
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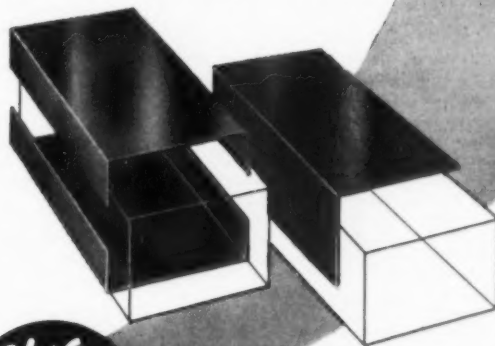


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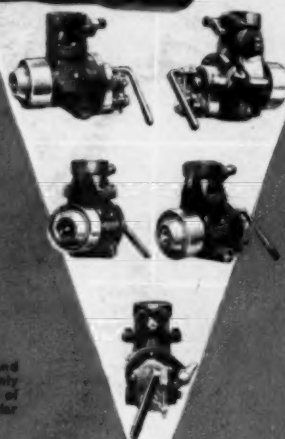
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PRN. 4 cap. 30 imp. gals/hr.

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NU-WAY HEATING PLANTS LTD. (BOX A 500) DROITWICH
and at LONDON, MANCHESTER, NEWCASTLE, GLASGOW, BELFAST, DUBLIN, BRISTOL.